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NONMETALS TEST AND EVALUATION

Delivery Order 0001: Alternate Positive Pressure Cure Cycles for 250 °F-Curing Epoxy Film Adhesives

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THIS TECHNICAL REPORT IS APPROVED FOR PUBLICATION.

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14. ABSTRACT (*Maximum 200 words*)
On-component bonded repairs routinely encounter temperature limitations due to safety considerations and the presence of vulnerable materials or systems in the repair vicinity, such as elastomers and wiring. Due to these limitations, engineers desire the capability to cure epoxy adhesives at temperatures lower than those recommended by the manufacturers. Cytec Engineered Materials' FM 73M and 3M Company's AF 163-2M as well as Henkel's Hysol EA 9628 and Hysol EA 9696 adhesives were evaluated using cure cycles at 200°F and 180°F. Alternate cure times were identified via differential scanning calorimetry to optimize curing at 200°F and 180°F. Tensile lap shear, floating roller peel, and climbing drum peel tests were used to evaluate the corresponding bond strengths. Results obtained in this study proved curing the adhesives at 200°F and 180°F was feasible but led to reduced adhesive mechanical properties.

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Adhesive Bonding, Epoxy, Film Adhesive, Differential Scanning Calorimetry, Lap Shear, Peel, Reduced Temperature Cure

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

PREFACE

This report covers work performed by AFRL/MLSA and the University of Dayton Research Institute (UDRI) from June 2002 until December 2005. This work funded AFRL/MLSA and conducted under contract F33615-00-D-5600 Delivery Order 0001. The work was administered under the direction of the Materials Integrity Branch of the Systems Support Division, Air Force Materials and Manufacturing Directorate, Air Force Research Laboratory, Wright-Patterson Air Force Base, Ohio. Mr. James J. Mazza was the contract monitor. The Air Force Technical Leads on this program were Mr. James Mazza and Dr. Brett Bolan. The UDRI Contract Program Manager was Mr. Roger Rondeau. The UDRI Technical Lead was Mr. Dan McCray.

Ms. Kara Storage is thanked for performing a portion of the differential scanning calorimetry testing. Ms. Molly Brown and Ms. Kelly Feirstine, Southwestern Ohio Council for Higher Education (SOCHE), are thanked for assistance performing the mechanical testing in this program.

1 BACKGROUND

Adhesive manufacturers provide recommended cure cycles for their adhesive materials. Several commonly used aerospace structural epoxy film adhesives have a recommended optimal cure cycle of 60 minutes at 250°F under 35-40 psi positive pressure. Examples include Cytec Engineered Materials' FM 73M, 3M Company's AF 163-2M and Henkel's products Hysol EA 9628 and Hysol EA 9696. These materials are commonly used at Air Force Air Logistic Centers (ALCs) for structural repair bonding. Although manufacturers recommend 60 minutes at 250°F to fully cure the above-mentioned adhesives, repaired aircraft components can have maximum temperature limitations below 250°F that prevent the recommended cure cycles from being utilized. ALC engineers would like the option of using lower-temperature cure cycles for these adhesives, despite the increase in cure time, as long as acceptable mechanical strengths can be obtained. Therefore, alternate cure cycles for these commonly used adhesives were defined at 200°F and 180°F. Results of mechanical testing at these alternate temperatures were compared to results obtained from specimens bonded using the manufacturers' recommended cure cycle.

2 TEST PROGRAM

Alternate cure cycles were defined and evaluated for the following 250°F-curing epoxy film adhesives: (1) FM 73M, 0.06 pounds per square foot (psf), (2) AF 163-2M, 0.06 psf, (3) EA 9628, 0.06 psf, and (4) EA 9696, 0.06 psf. Initially, differential scanning calorimetry (DSC) was performed on each adhesive to define alternate cure times for temperatures of 180°F and 200°F. Once the alternate cure cycles were defined, mechanical testing was performed on specimens cured using these alternate cure cycles to determine the effects of the alternate cure cycles on bond strength.

2.1 Differential Scanning Calorimetry (DSC) Testing

DSC is a common thermal analysis technique utilized to measure the temperature/heat flow associated with transitions (e.g. glass transition temperature (T_g), melting, crystallization) and chemical changes (e.g. polymerization) in polymeric materials as a function of temperature and time[1].

A DSC instrument measures the amount of energy required to maintain a sample at a programmed temperature. For uncured epoxy-based adhesives, as the temperature increases, the material undergoes polymerization, generating heat due to the exothermic nature of the chemical reactions occurring. This is displayed by the DSC instrument as a peak in a heat flow versus temperature curve, the area under which represents the energy generated during the curing process. If a sample is only partially polymerized (e.g. due to a lower temperature cure cycle), then only a portion of the energy will be released (seen as a smaller peak). Thus, the heat released during a DSC test can be used as a measure of the degree of cure.

For this study, a TA Instruments DSC model Q100 was used to investigate the effect of the alternate cure schedules on the thermo-physical properties of the specified adhesives. A testing rate of 10°C/min was used for all dynamic scans, including the baseline determination for each adhesive. It was assumed, for all adhesives except FM 73M, the measured exotherms obtained during baseline runs represented the energy released for a fully (100%) cured adhesive. However, for FM 73M, the energy evolved during the one-hour isothermal cure at 250°F (i.e.

$\Delta H_{isothermal}$ = 216 J/g from Table 2) was taken to represent the energy released for a fully (100%) cured adhesive. This was necessitated because the dynamic baseline run yielded a lower total exotherm value (203 J/g), possibly as a result of different/preferred chemical reactions occurring during this cure cycle. The FM 73M adhesive was beyond its stated shelf-life (although continually stored below -20°F), and this could be a contributor to the observed phenomenon as well. This will be further investigated in the future.

For the alternate cure cycles, the resins were rapidly heated to the isothermal hold temperature (in less than 3 minutes) and, therefore, it was not believed any measurably significant chemical reaction occurred during this time. In analyzing the alternate cure cycles, the areas under the curve (heat flow versus temperature) during the isothermal segment were quantified and compared to the results obtained from the baseline run for that adhesive. The ratio of these two quantities represents the extent of polymerization (curing) that occurred during the alternate cure cycle. For example:

$$\frac{\text{Alternate cure (EA 9696-8 hours @ 180°F)}}{\text{Baseline (EA 9696)}} = \frac{\Delta H_{isothermal}}{\Delta H_R} = \frac{168 \text{ J/g}}{231 \text{ J/g}} \sim 73\%$$

This denotes that an 8-hour cure at 180°F results in EA 9696 adhesive that is approximately 73% cured.

After each baseline and alternate cure cycle experiment, the samples were rapidly cooled to room temperature (70°F) then dynamically scanned at 10°C/min to determine the residual heat of reaction ($\Delta H_{residual}$) as well as the materials' glass transition temperatures (T_g). Throughout this study, T_g was determined as the inflection point (I) of the thermal curve.

2.2 Mechanical Testing

In order to determine the effect of altering the cure cycle on adhesive bond strength, tensile lap shear[2], floating roller peel[3], and honeycomb climbing drum peel[4] tests were conducted. Adherends used for lap shear and floating roller peel testing were composed of bare Al 2024-T3 that were phosphoric acid anodized[5] and primed with Cytec Engineered Materials BR 127 bond primer. Climbing drum peel specimens were fabricated from bare Al 2024-T3 face sheets bonded to 7.9-1/4-40(5052) core (MIL-C-7438[6]). The climbing drum peel facesheets were treated using P2 paste acid etch and primed with BR 127 primer. The P2 paste acid etch process followed the P2 tank process[7] except the acid was mixed with Cab-O-Sil silica filler (130 mL acid:14.75 g Cab-O-Sil), applied via a brush for 25 minutes, then rinsed with water. The aluminum core was cleaned with an alkaline cleaner and rinsed with water prior to bonding. BR 127 primer was applied via a Binks 105 spray gun to a nominal thickness of 0.2 mil (0.0002 inch), dried for 30 minutes at ambient laboratory temperature (70°F), then cured for 60 minutes at 250°F in an air-circulating oven.

The matrix for mechanical testing is shown in Table 1. Specimens were bonded with each adhesive using alternate cure cycles derived from the DSC test results in Table 2. Actual cure times varied for each adhesive and were based on the apparent time at which cure was completed (Table 2) with additional time added to ensure cure was completed. Test results obtained with the alternate cures were compared to results obtained using the manufacturers' recommended cure cycle of 60 minutes at 250°F. All bonding was conducted in a portable autoclave (lap shear and floating roller peel specimens) or hydraulic press (climbing drum peel specimens) using 35 psi positive pressure. Test panels were machined into specimens and tested according to Table 1. Dry specimens were tested after a 10-minute exposure to temperature. Wet specimens were tested after 4-minute exposure to temperature.

Table 1: Mechanical Test Matrix for the Evaluation of Alternate Cure Cycles

Adhesive	Cure Cycles	Number of Specimens per Condition					
		Lap Shear			Roller Peel		Climb Drum Peel
		70°F	180°F-dry	180°F-wet*	-65°F	70°F	70°F
EA 9628	1 hr @ 250°F	5	5	5	5	5	6
	4 hr @ 200°F	5	5	5	5	5	6
	8 hr @ 180°F	5	5	5	5	5	6
EA 9696	1 hr @ 250°F	5	5	5	5	5	6
	6 hr @ 200°F	5	5	5	5	5	6
	8 hr @ 180°F	5	5	5	5	5	6
FM 73M	1 hr @ 250°F	5	5	5	5	5	6
	4 hr @ 200°F	5	5	5	5	5	6
	7 hr @ 180°F	5	5	5	5	5	6
AF 163-2M	1 hr @ 250°F	5	5	5	5	5	6
	6 hr @ 200°F	5	5	5	5	5	6
	8 hr @ 180°F	5	5	5	5	5	6

Note: * "wet" specimens were exposed to 140°F and 98% RH for 60 days prior to testing

3 RESULTS

Summarized results of the DSC and mechanical testing are shown in this section. A complete record of the DSC scans is presented in Appendix A. Complete results of the mechanical testing are shown in Appendix B.

3.1 Differential Scanning Calorimetry (DSC) Testing

Summarized results of the DSC testing are shown in Table 2. In general, the adhesives evaluated were found to exhibit similar thermal curing characteristics; longer times at lower temperatures were required to achieve a cured state (albeit with lower percent chemical conversion). At a cure temperature of 200°F, all four adhesives achieved similar cure states (i.e. T_g and % cure close to those obtained following the manufacturer's recommended cure schedule of one hour at 250°F), but with FM 73M accomplishing this in significantly less time. For a cure temperature of 180°F, FM 73M appears to not only have the ability to cure more quickly but also more fully than the other three adhesives.

3.2 Mechanical Testing

Results of the mechanical testing are shown in Table 3. All failure modes are cohesive (within the adhesive layer) unless otherwise noted. Mixed failure modes were a combination of adhesive and cohesive failures. Adhesive failures occurred at the primer-adhesive interface. Tensile lap shear results do not appear to be drastically reduced due to the reduced cure temperatures. However, floating roller peel strength and climbing drum peel torques appear to be reduced due to the lower-temperature cures. The extents of the strength and torque reductions are discussed in more depth in Section 3.3.

Table 2: DSC Results for AF 163-2M, EA 9696, EA 9628 and FM 73M

Cure Cycle	Property	AF 163-2M	EA 9696	EA 9628	FM 73M
Baseline (10°C/min to 250°C (482°F))	Heat of Reaction from Dynamic DSC test (ΔH_R = J/g)	176	231	272	203 [4]
	Resultant T_g	214°F (101°C)	216°F (102°C)	225°F (107°C)	165°F (74°C)[4]
1hr @ 250°F (121°C)	Energy evolved during isothermal hold ($\Delta H_{isothermal}$ = J/g)[1]	179	231	275	216 [3]
	Apparent time at which cure was completed (min)[2]	33	25	27	35
	T_g	234°F (112°C)	239°F (115°C)	243°F (117°C)	201°F (94°C)
	Residual heat of reaction after isothermal cure ($\Delta H_{residual}$ = J/g)	5	0	0	3
	% cure [($\Delta H_{isothermal}/\Delta H_R$)*100] = J/g	97	100	100	99+
6 hrs @ 200°F (93°C)	Energy evolved during isothermal hold ($\Delta H_{isothermal}$ = J/g)[1]	157	209	241	199
	Apparent time at which cure was completed (min)[2]	204	179	158	113
	T_g	234°F (112°C)	232°F (111°C)	235°F (113°C)	228°F (109°C)
	Residual heat of reaction after isothermal cure ($\Delta H_{residual}$ = J/g)	15	25	29	21
	% cure [($\Delta H_{isothermal}/\Delta H_R$)*100] = J/g	89	90	89	92
8 hrs @ 180°F (82°C)	Energy evolved during isothermal hold ($\Delta H_{isothermal}$ = J/g)[1]	134	168	189	174
	Apparent time at which cure was completed (min)[2]	350	350	295	216
	T_g	207°F (97°C)	208°F (98°C)	214°F (101°C)	212°F (100°C)
	Residual heat of reaction after isothermal cure ($\Delta H_{residual}$ = J/g)	31	47	46	35
	% cure [($\Delta H_{isothermal}/\Delta H_R$)*100] = J/g	76	73	69	81

<u>Notes</u>

1 These values are estimates and subject to errors of up to 3%. This is attributable to the uncertainty in determining the precise time at which significant amounts of chemical reactions begin and end (i.e. where to place the markers for analyzing the area under the "peak"). In the isothermal mode of operation, the DSC instrument becomes less sensitive to chemical reactions that may be occurring at slow rates over long periods of time.

2 Completion times are estimated as the amount of time to achieve 90% of the maximum chemical conversion level for the particular cure condition. The completion times are subject to the same errors (with the same rationale) as those mentioned in Note 1.

3 For FM 73M, it was assumed the energy evolved during the isothermal cure of 1 hour at 250°F (i.e. $\Delta H_{isothermal}$ = 216 J/g) represents the energy released for a fully (100%) cured adhesive and not that evolved during the baseline dynamic DSC test (i.e. ΔH_R = 203 J/g).

4 These values are below those claimed by the manufacture, which might be attributed to the adhesive being beyond its stated shelf life (despite being constantly stored at or below -20°F).

Table 3: Mechanical Test Results

Adhesive	Cure Cycles	Average Strength					
		Lap Shear (psi)			Roller Peel (pli)		Climb Drum Peel (in*lb/in torque)
		70°F	180°F-dry	180°F-wet*	-65°F	70°F	70°F
EA 9628	1 hr @ 250°F	5915	4102	3516	53.4	57.9	17.6
	4 hr @ 200°F	5609	4561	3593	35.6	36.5	11.2
	8 hr @ 180°F	5057	3616	3501	33.9	30.6	8.8
EA 9696	1 hr @ 250°F	5620	4798	3198	56.5	71.9	20.7
	6 hr @ 200°F	5906	4253	3743	52.3	57.2	13.5
	8 hr @ 180°F	5465	3534	3614	46.3	48.4	12.9
FM 73M	1 hr @ 250°F	5788	4329	2891^{m}	68.1	88.9	22.9
	4 hr @ 200°F	6132	4162	3444^{m}	36.0	47.7	14.1
	7 hr @ 180°F	6171	4203	3503^{m}	31.1^{m}	42.6	11.8
AF 163-2M	1 hr @ 250°F	5704	3805	2829	68.8^{m}	77.6	18.0
	6 hr @ 200°F	5665	3786	3024	59.4^{m}	64.1	15.1
	8 hr @ 180°F	5236	2838^{m}	2985	54.3	62.0	15.6

* "wet" specimens were exposed to 140°F and 98% RH for 60 days prior to testing

^{m} specimens exhibited a mix of cohesive and adhesive failure modes

3.3 Effect of Reduced-Temperature Cures on Bond Strength

The main concern with using a low-temperature alternate cure cycle is the possibility of losing bond strength. In order to assess the effects of using the alternate cure cycles, the mechanical strengths from Table 3 were used to generate normalized strength charts for each adhesive to determine the knockdown for each mechanical test and testing condition due to the alternate cure cycle. Normalized strengths were determined for each test condition with each adhesive using the following equation:

$$\text{Normalized strength (\%)} = (\text{Strength}_{\text{alternate cure}}) / (\text{Strength}_{\text{1 hr @ 250°F cure}}).$$

The normalized strength chart for EA 9628 is shown in Figure 1. Curing the adhesive at 200°F or 180°F did not adversely affect lap shear strength under any conditions. However, the lower-temperature cure cycles produced reduced floating roller peel strengths and climbing drum peel torques. Curing at 180°F reduced peel strengths (~50-60% of original strength) more than curing at 200°F.

The normalized strength chart for EA 9696 is shown in Figure 2. Curing the adhesive at 200°F or 180°F did not adversely affect lap shear strength at RT or 180°F-wet. However, there was a reduction in 180°F-dry lap shear strength due to low-temperature curing. The lower-temperature cure cycles produced slight reductions in -65°F floating roller peel strength. Floating roller peel testing and climbing drum peel testing conducted at 70°F showed reduced bond strengths and torques due to lower-temperature curing. As with EA 9628, curing at 180°F reduced the bond strengths more than did curing at 200°F.

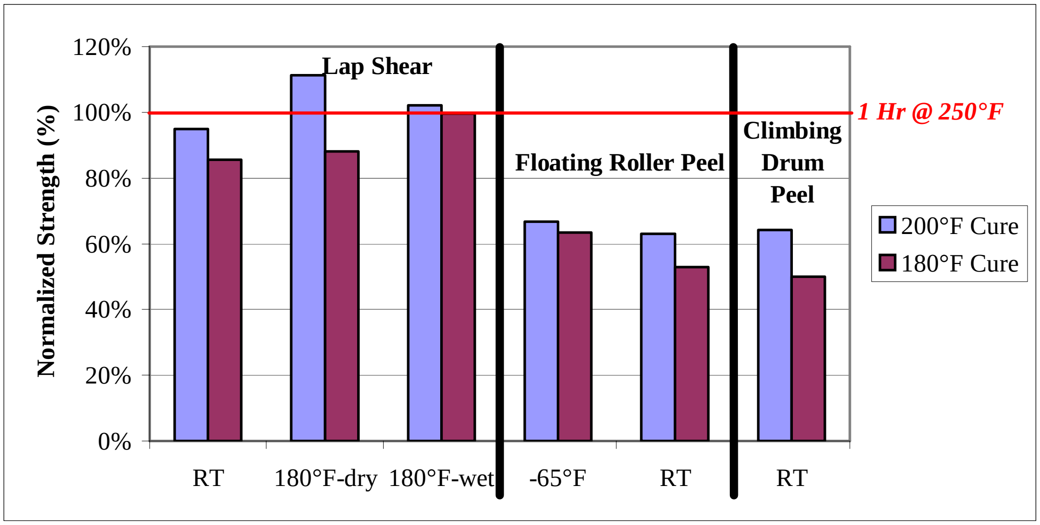


Figure 1: Effect of Alternate Cure Cycle on Normalized Strength for EA 9628

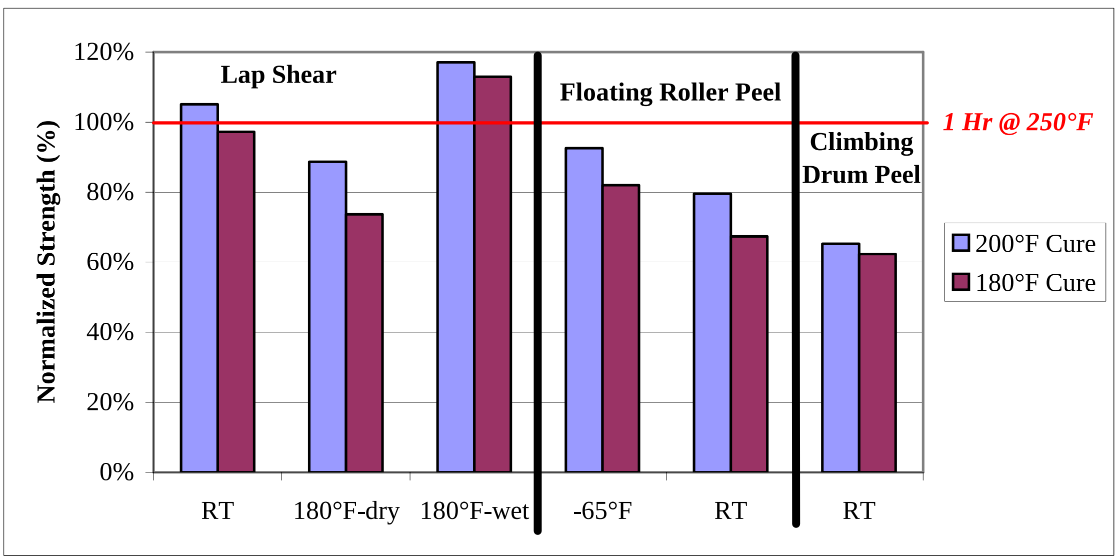


Figure 2: Effect of Alternate Cure Cycle on Normalized Strength for EA 9696

The normalized strength chart for FM 73M is shown in Figure 3. Curing the adhesive at 200°F or 180°F did not adversely affect lap shear strengths under any conditions. There was a substantial reduction in both floating roller peel strength and climbing drum peel torque due to reducing the cure temperature.

The normalized strength chart for AF 163-2M is shown in Figure 4. Curing the adhesive at 200°F or 180°F did not adversely affect lap shear strength at RT or 180°F-wet, although 180°F-dry lap shear strength was reduced due to low-temperature curing. Unlike the other film adhesives, AF 163-2M exhibited a smaller reduction in floating roller peel strength and climbing drum peel torque due to lower-temperature curing.

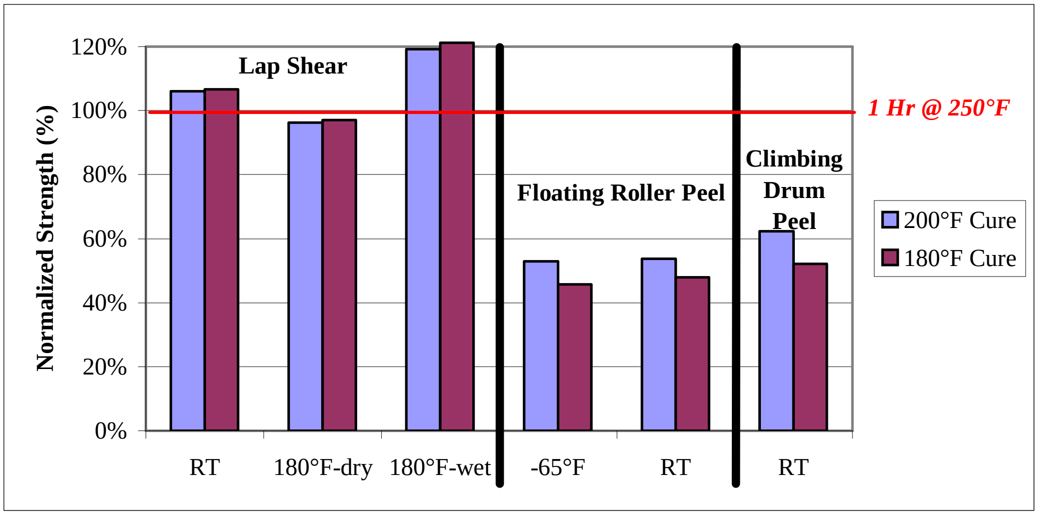


Figure 3: Effect of Alternate Cure Cycle on Normalized Strength for FM 73M

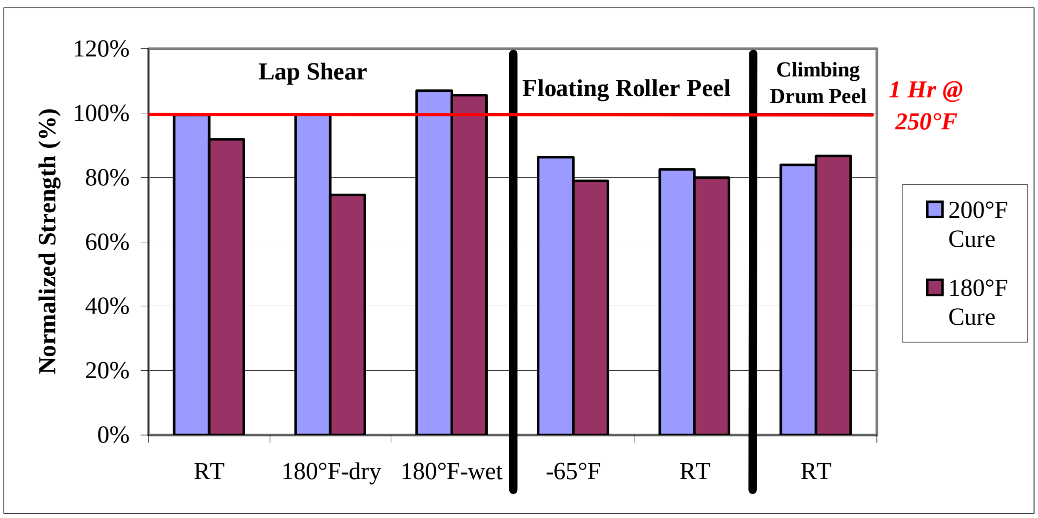


Figure 4: Effect of Alternate Cure Cycle on Normalized Strength for AF 163-2M

When comparing Figures 1-4, it appears AF 163-2M retained floating roller peel and climbing drum peel properties better than the other adhesives when cured at 200°F and 180°F. On the other end of the spectrum, FM 73M appeared to lose peel properties more easily than the other evaluated adhesives when cured at 200°F and 180°F, even though FM 73M had a higher percent cure as determined by DSC.

4 CONCLUSIONS

DSC data evaluating alternate low-temperature cures were generated on four commonly used 250°F-cure epoxy film adhesives: EA 9628, EA 9696, FM 73M and AF 163-2M. According to DSC results, curing the adhesives at 200°F for 6 hours provided thermo-physical properties (T_g and degree of cure) similar to those obtained when using the manufacturers' recommended cure temperature of 250°F for one hour. Curing at 180°F provided properties substantially less than those obtained using the 250°F cure temperature.

Tensile lap shear, floating roller peel, and climbing drum peel specimens were fabricated using the evaluated adhesives with 250°F, 200°F, and 180°F cure temperatures. Tensile lap shear strength was not appreciably reduced for any of the adhesives cured at 200°F. Reduction in lap shear strength was observed for EA 9628, EA 9696, and AF 163-2M when testing at elevated temperature after curing at 180°F. Floating roller peel and climbing drum peel test results were significantly reduced due to curing at temperatures lower than the recommended 250°F. Peel strengths obtained when curing FM 73M and EA 9628 at 200°F & 180°F were reduced by 40-50% when compared to peel strengths obtained using a 250°F cure. EA 9696 and AF 163-2M fared better when cured at 180°F & 200°F, experiencing peel strength losses around 20-30% when compared to specimens cured at 250°F.

Although curing the evaluated adhesives at 200°F instead of 250°F appears to provide acceptable results, it should be noted this adhesive evaluation was not comprehensive. This evaluation was meant as an initial assessment of reduced-temperature curing of typical “250°F-curing” modified epoxy film adhesives. No work was performed to evaluate the effects of reduced-temperature cures on adhesive characteristics such as chemical resistance (e.g. jet fuel, hydraulic fluid, solvents, etc.), bond durability, or stress/strain relationships. All of these effects should be understood prior to implementing reduced-temperature cures for bonded structures.

REFERENCE

[1] ASTM E 1356-03, “Standard Test Method for Assignment of the Glass Transition Temperatures by Differential Scanning Calorimetry,” 2003.

2 ASTM D 1002-05, “Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal),” 2005.

[3] ASTM D 3167-03a (Reapproved 2004), “Standard Test Method for Floating Roller Peel Resistance of Adhesives,” 2004.

[4] ASTM D 1781-98 (Reapproved 2004), “Standard Method for Climbing Drum Peel Test for Adhesives,” Annual Book of ASTM Standards, Volume 15.06: Adhesives, 2004.

[5] ASTM D 3933-98 (Reapproved 2004), “Standard Guide for Preparation of Aluminum Surfaces for Structural Adhesives Bonding (Phosphoric Acid Anodizing),” 2004.

[6] MIL-C-7438F, “Military Specification for Core Material, Aluminum, for Sandwich Construction,” 13 March 1972.

[7] ASTM E 864-03, “Standard Practice for Surface Preparation of Aluminum Alloys to be Adhesively Bonded in Honeycomb Shelter Panels,” 2003.

APPENDIX A

Differential Scanning Calorimetry (DSC) Scans

Figure A1: Energy Released During Baseline Scan (10°C/min) for AF 163-2M

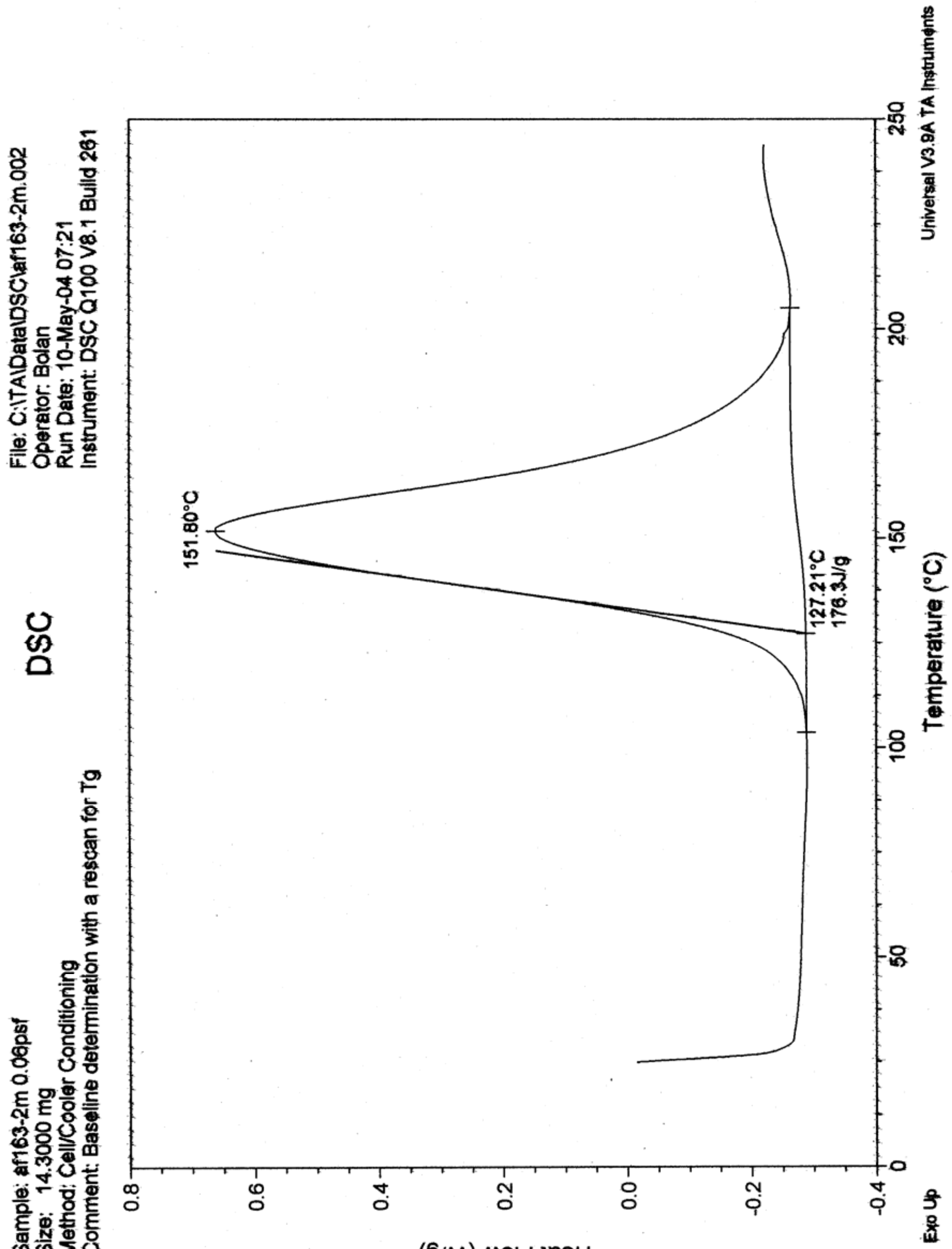


Figure A2: T_g of AF 163-2M After Baseline Scan

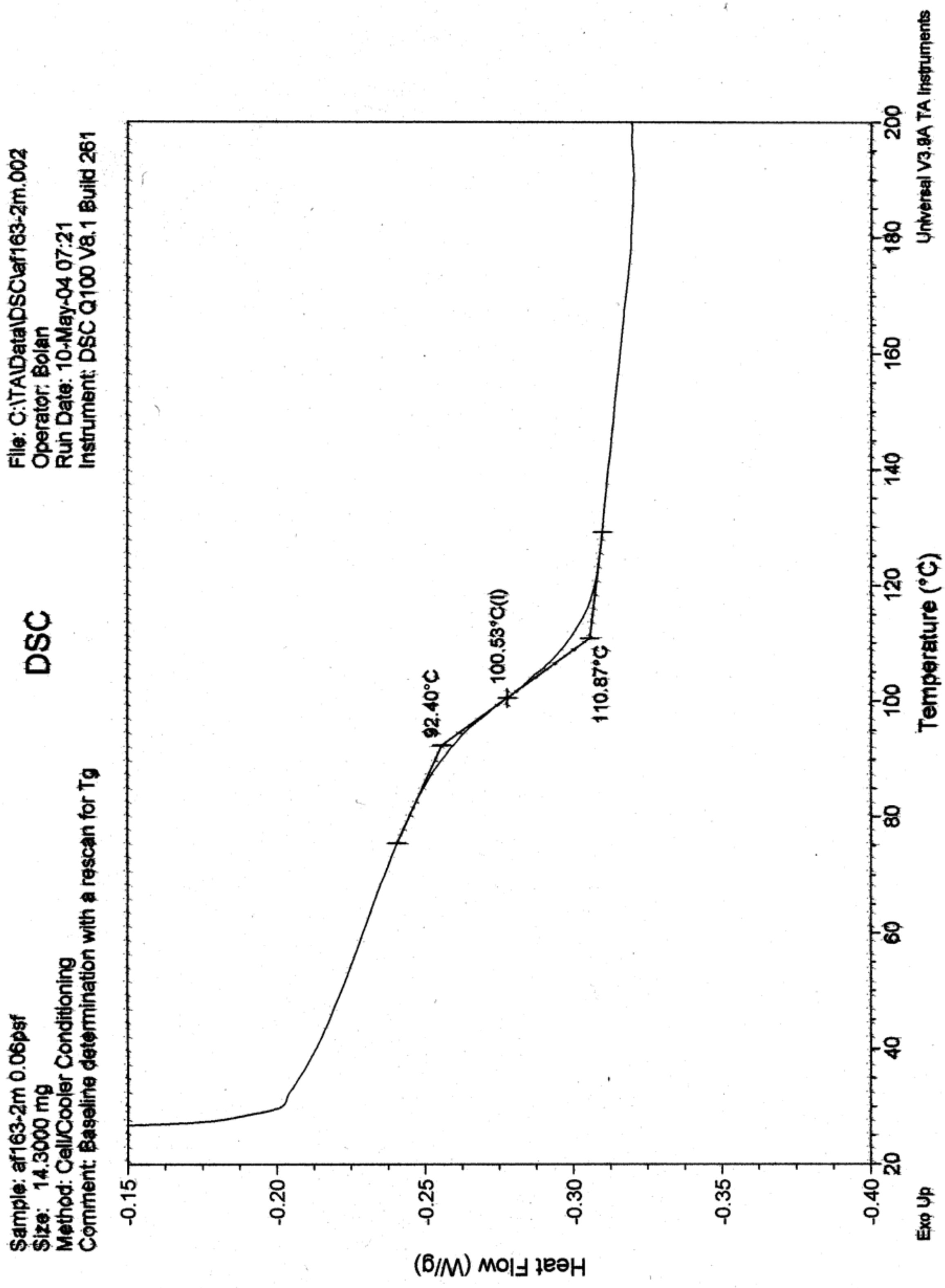


Figure A3: Energy Released During 1-Hour Cure at 250°F for AF 163-2M

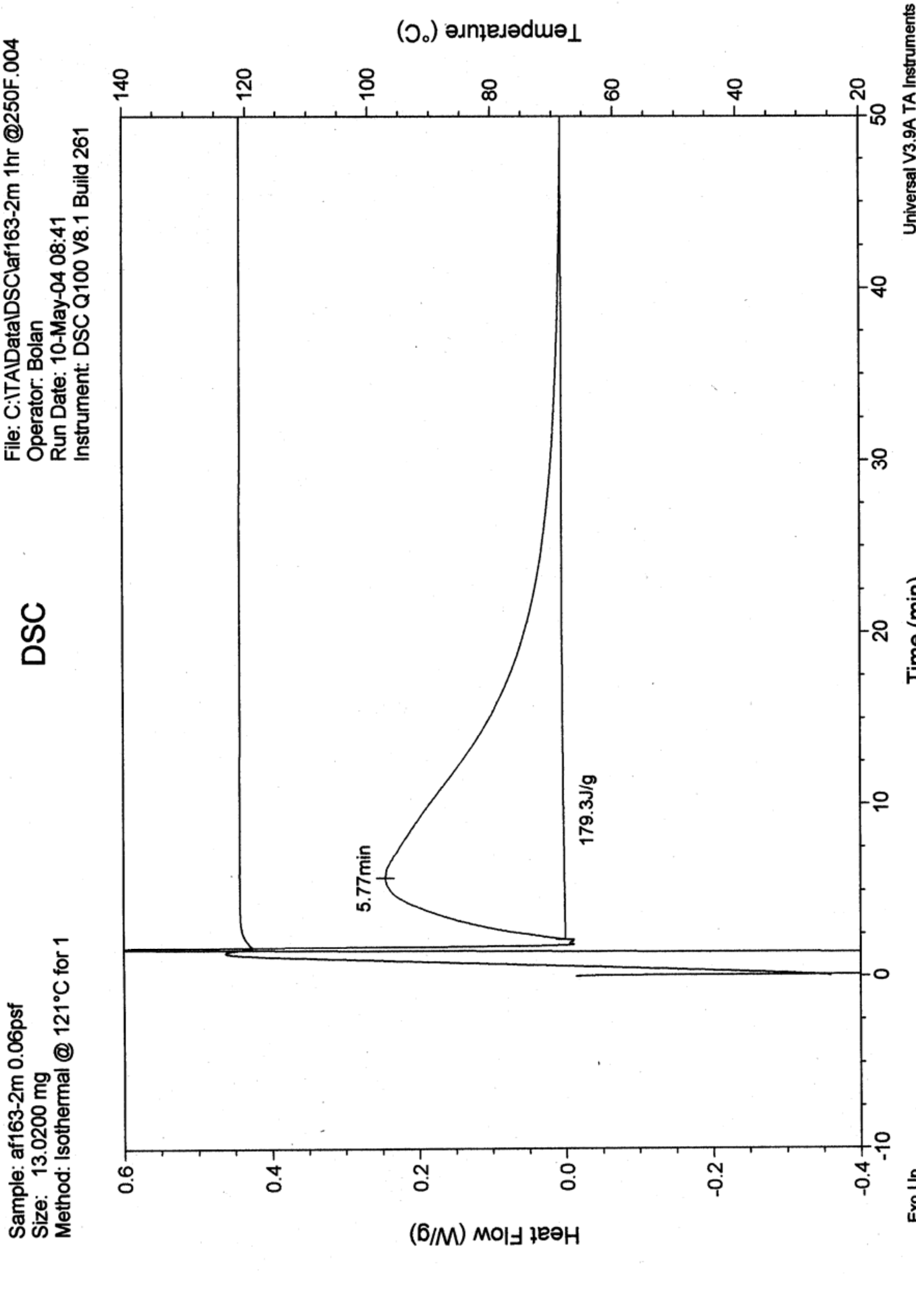


Figure A4: T_g and Residual Exotherm After 1 Hour at 250°F for AF 163-2M

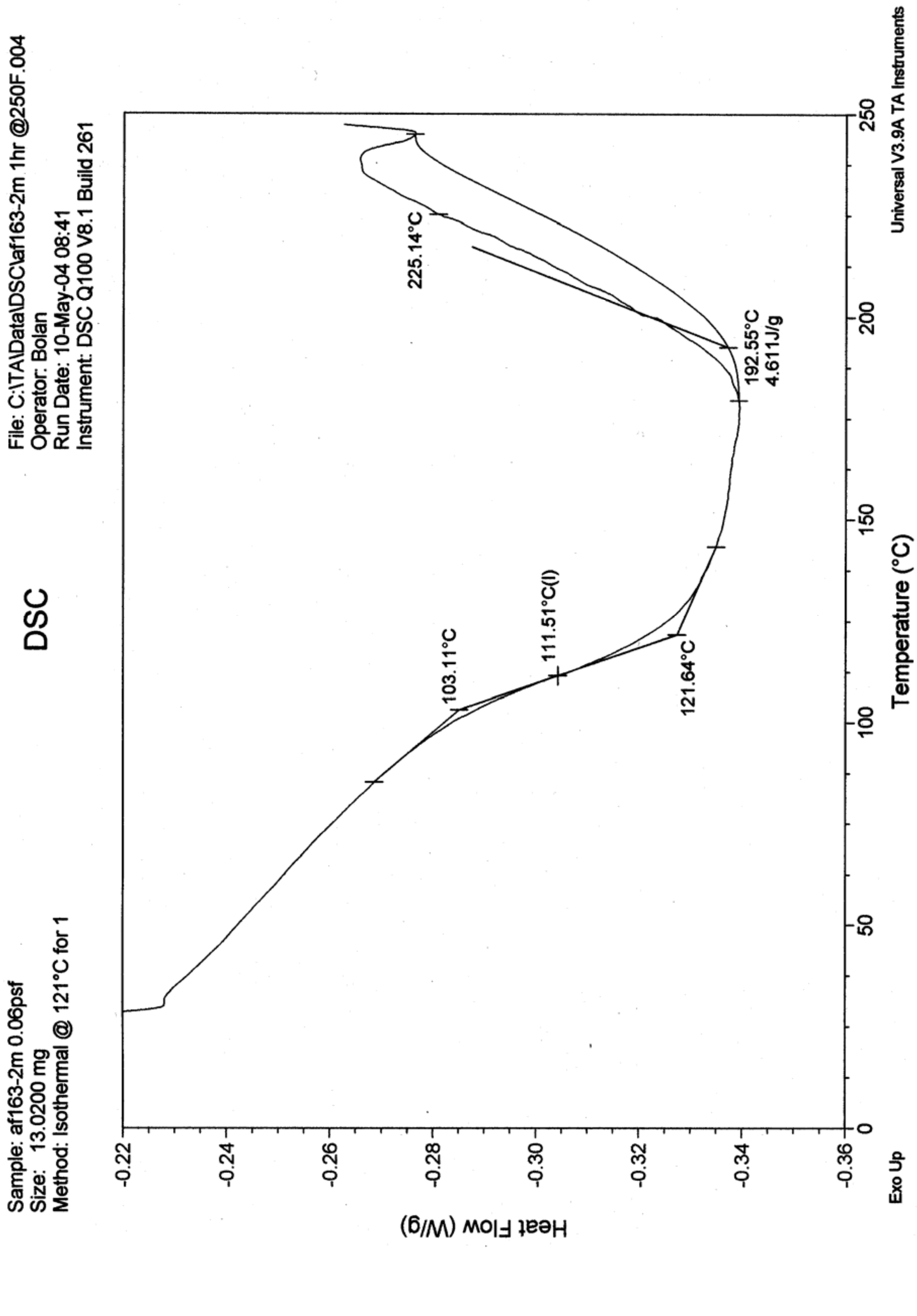


Figure A5: Energy Released During 6-Hour Cure at 200°F for AF 163-2M

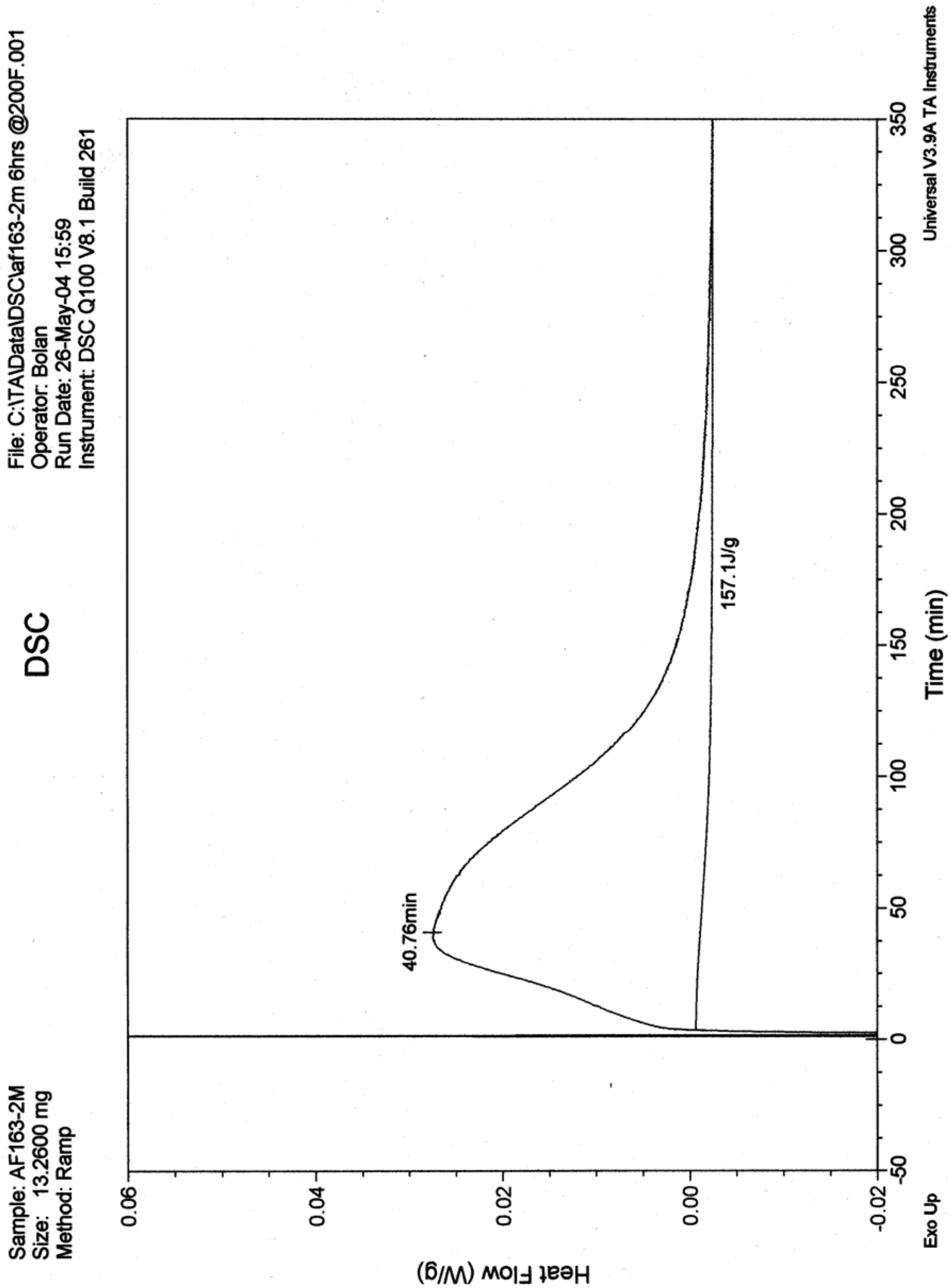


Figure A6: T_g and Residual Exotherm After 6 Hours at 200°F for AF 163-2M

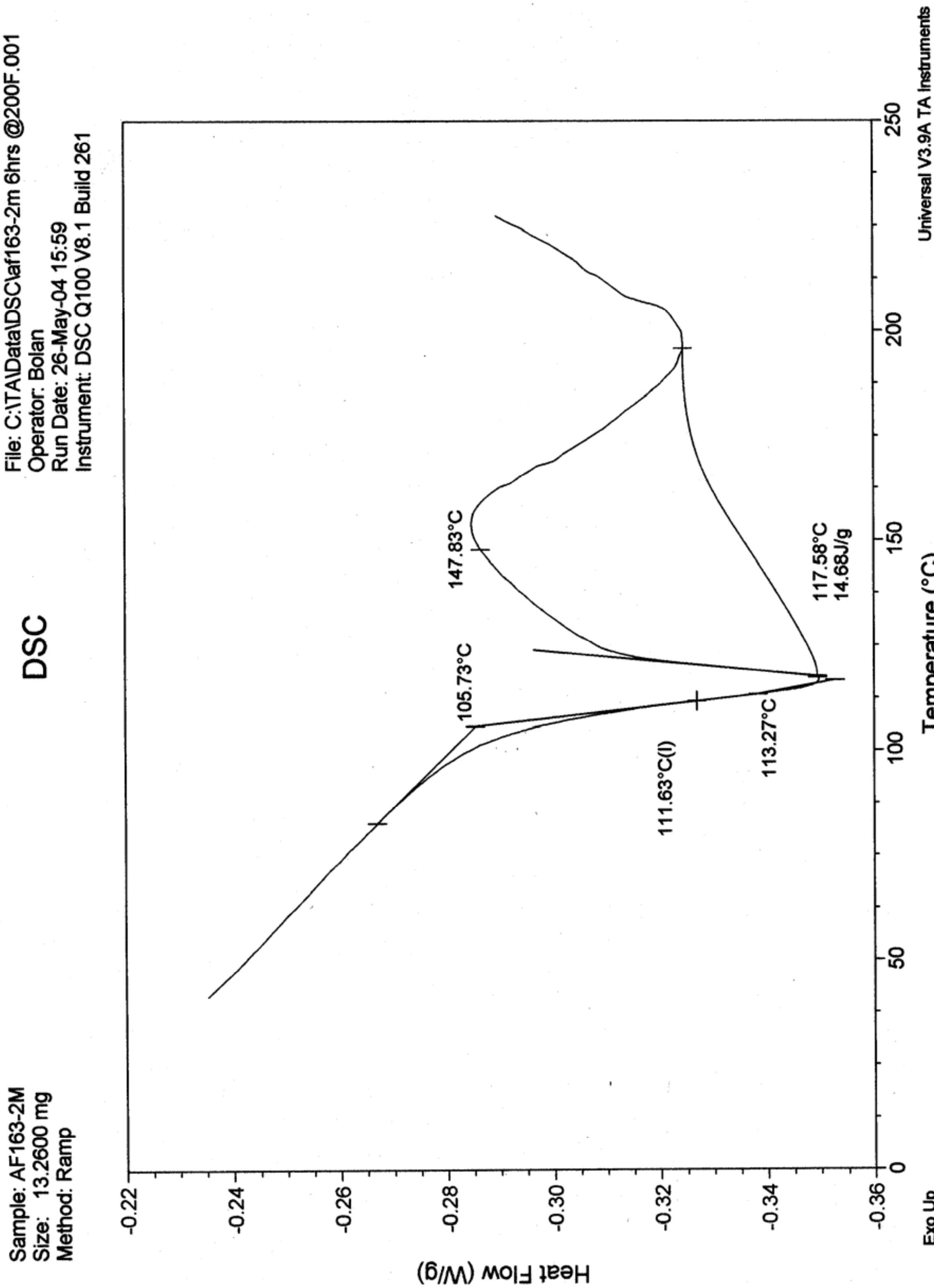


Figure A7: Energy Released During 8-Hour Cure at 180°F for AF 163-2M

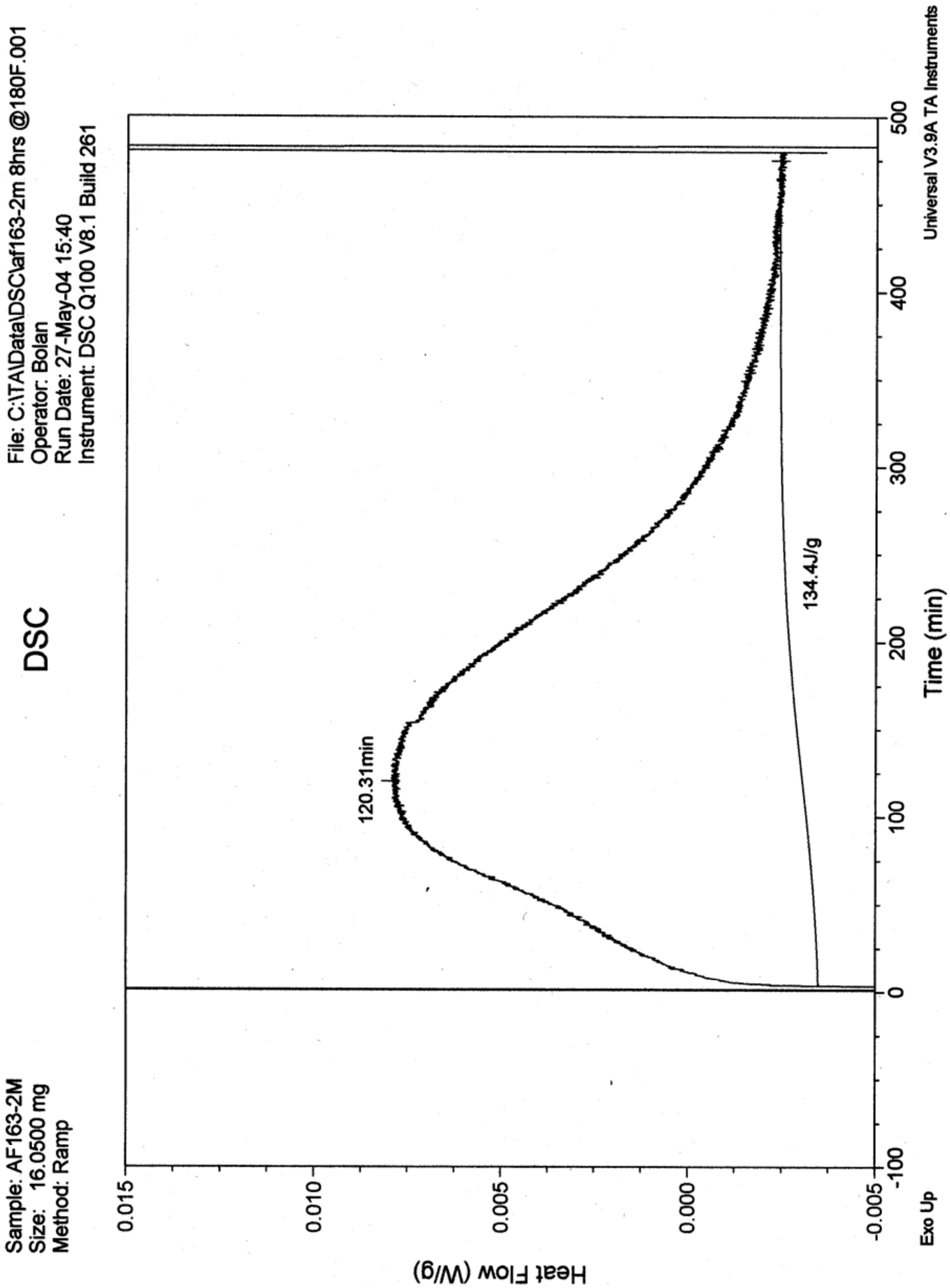


Figure A8: T_g and Residual Exotherm After 8 Hours at 180°F for AF 163-2M

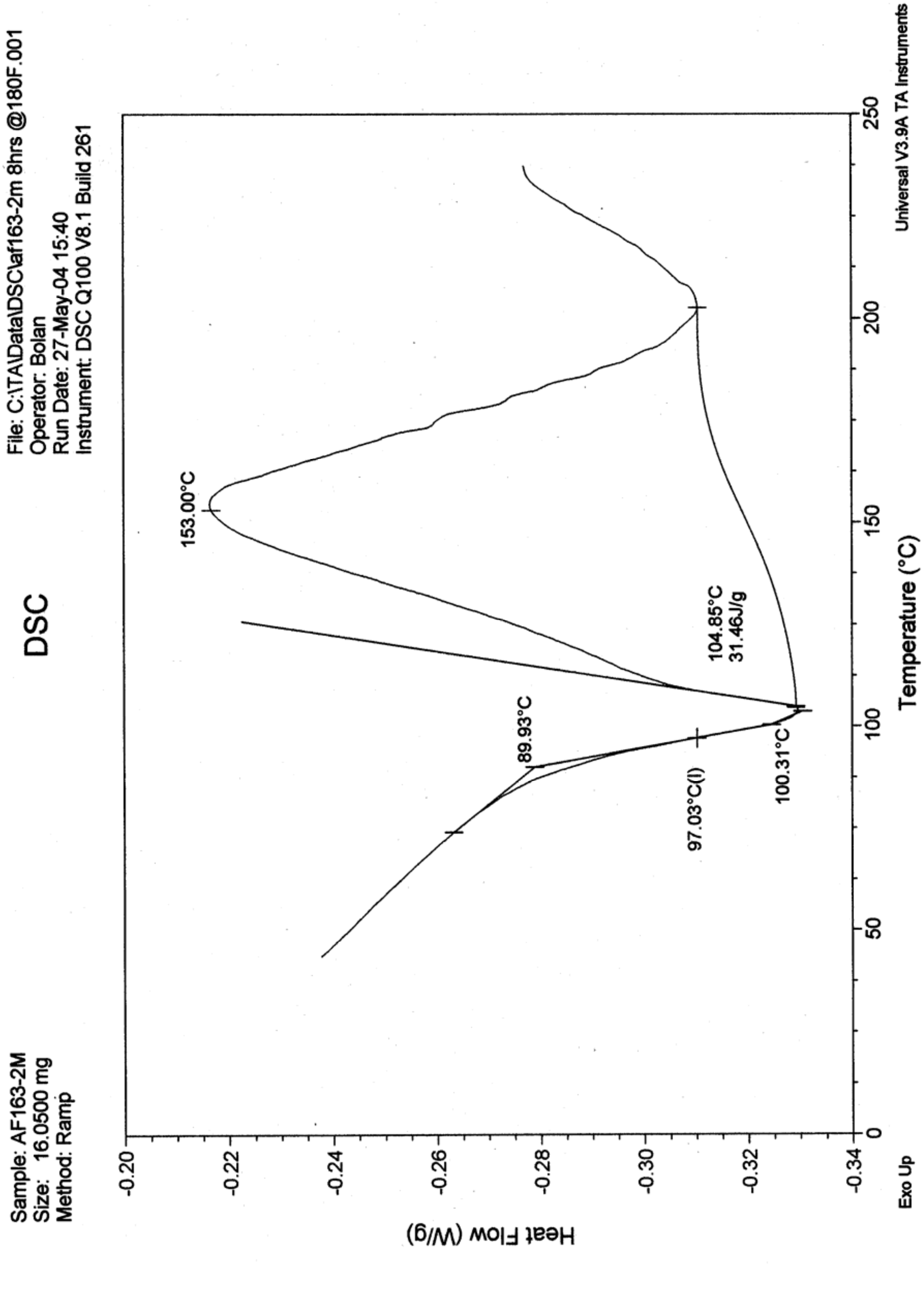


Figure A9: Energy Released During Baseline Scan (10°C/min) for EA 9696

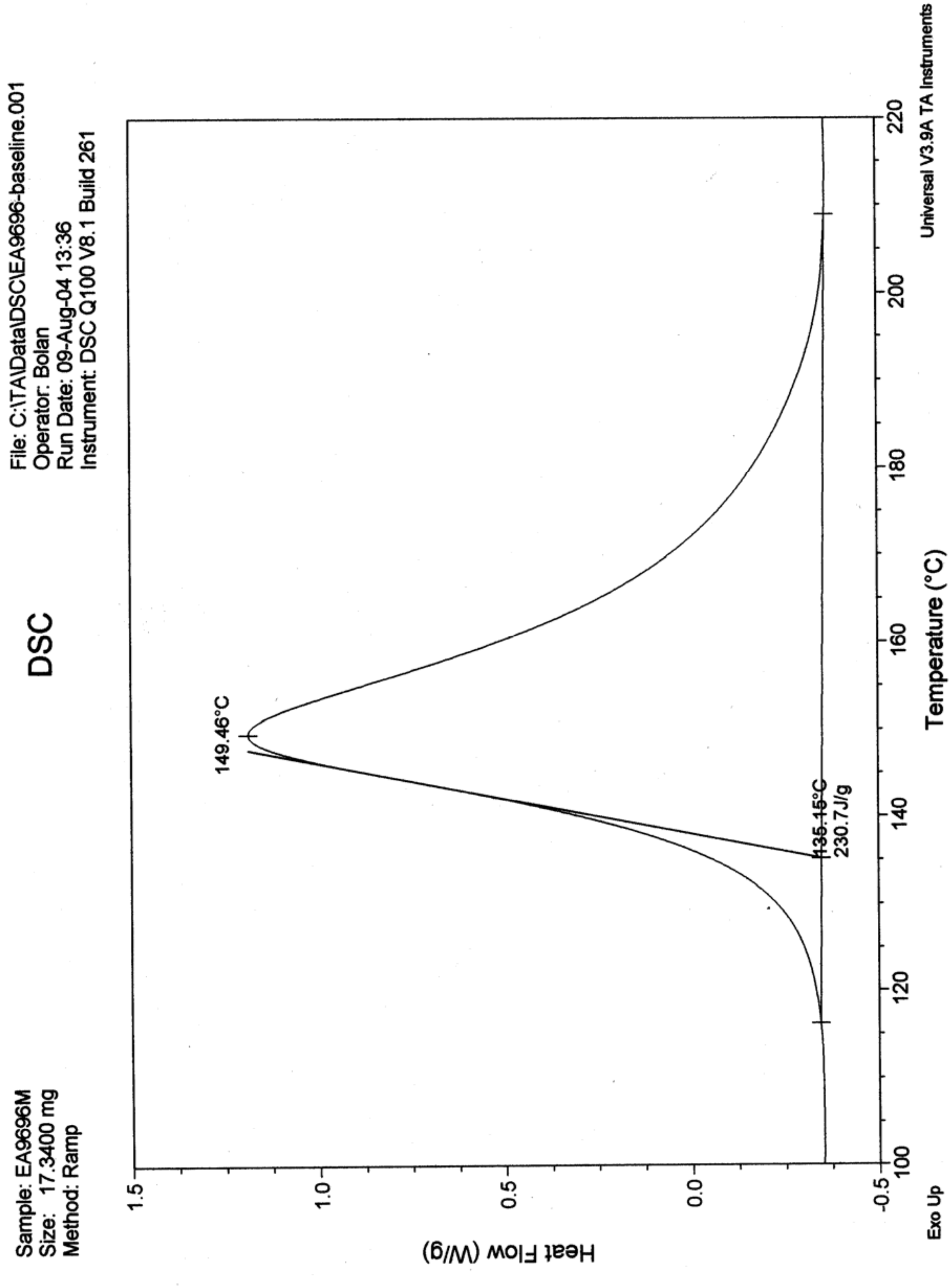


Figure A10: T_g of EA 9696 After Baseline Scan

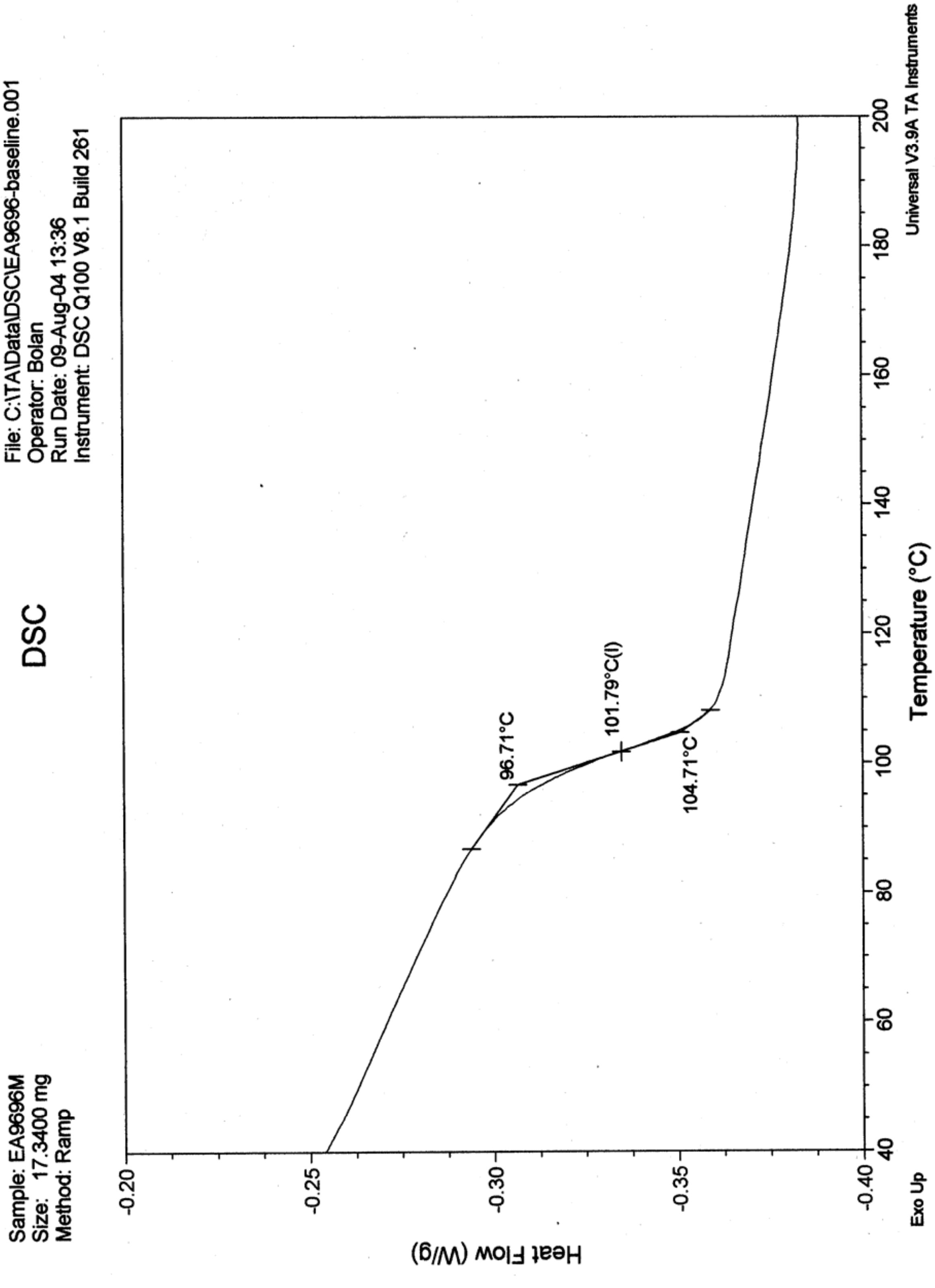


Figure A11: Energy Released During 1-Hour Cure at 250°F for EA 9696

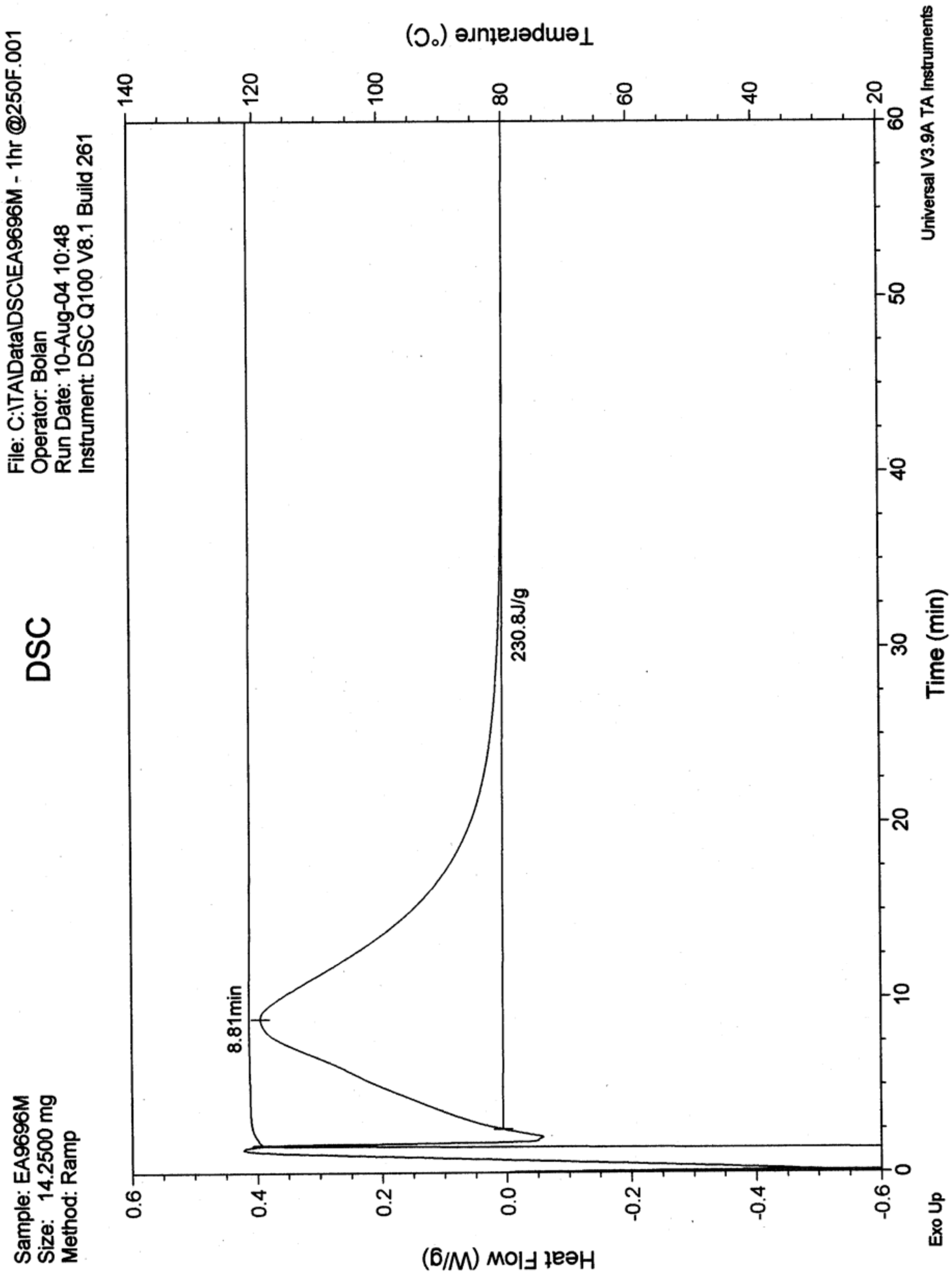


Figure A12: T_g and Residual Exotherm After 1 Hour at 250°F for EA 9696

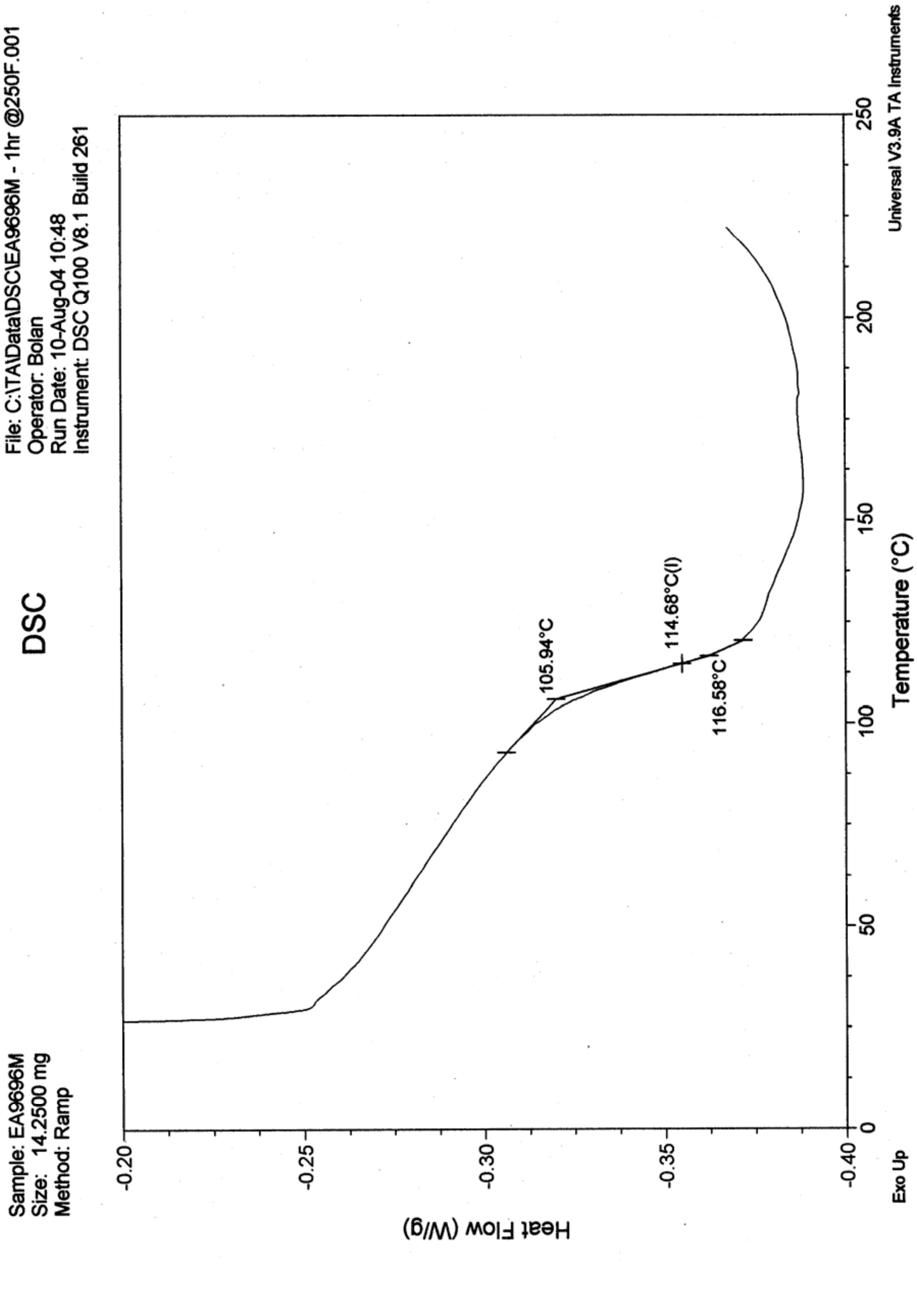


Figure A13: Energy Released During 6-Hour Cure at 200°F for EA 9696

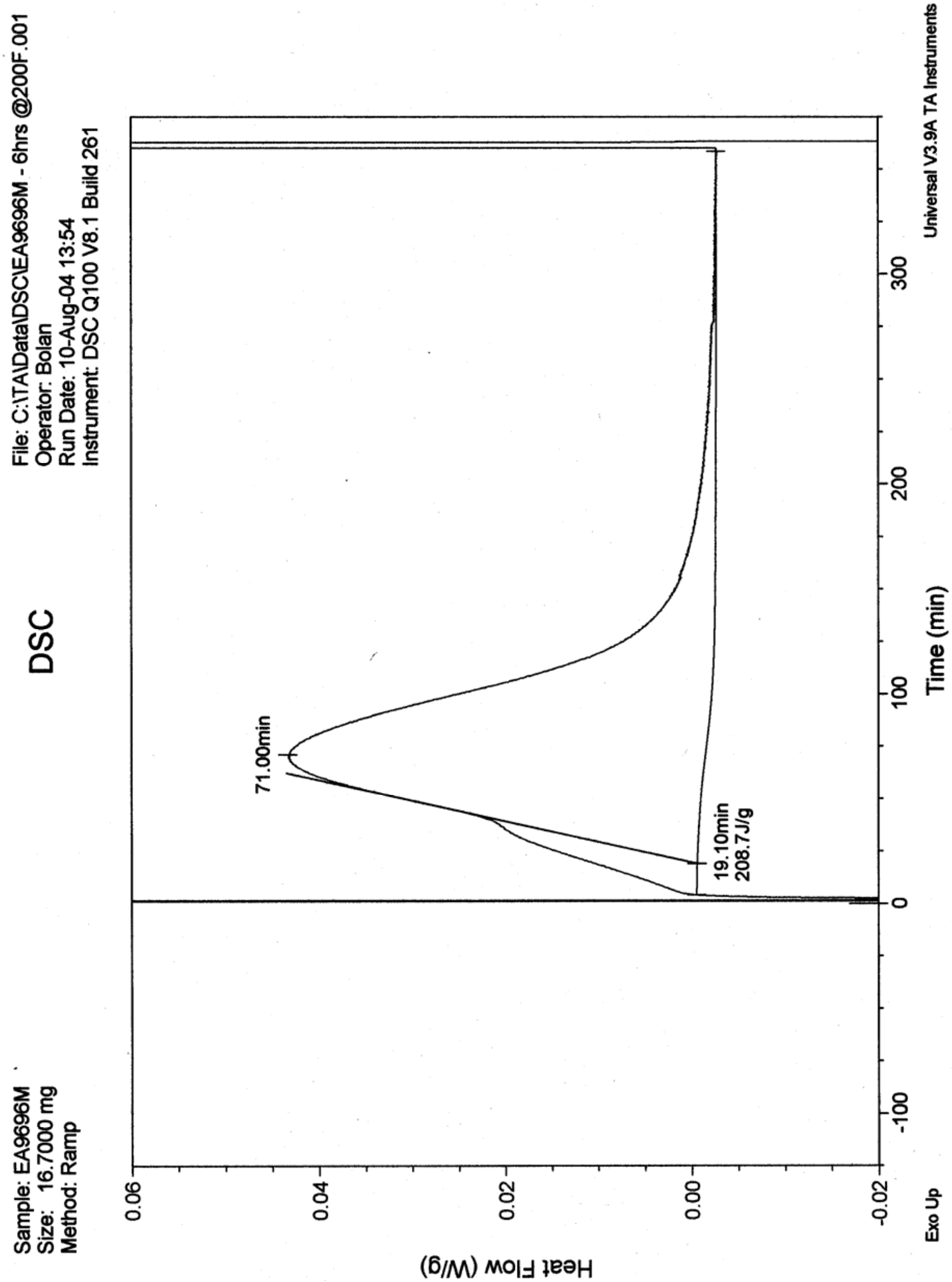


Figure A14: T_g and Residual Exotherm After 6 Hours at 200°F for EA 9696

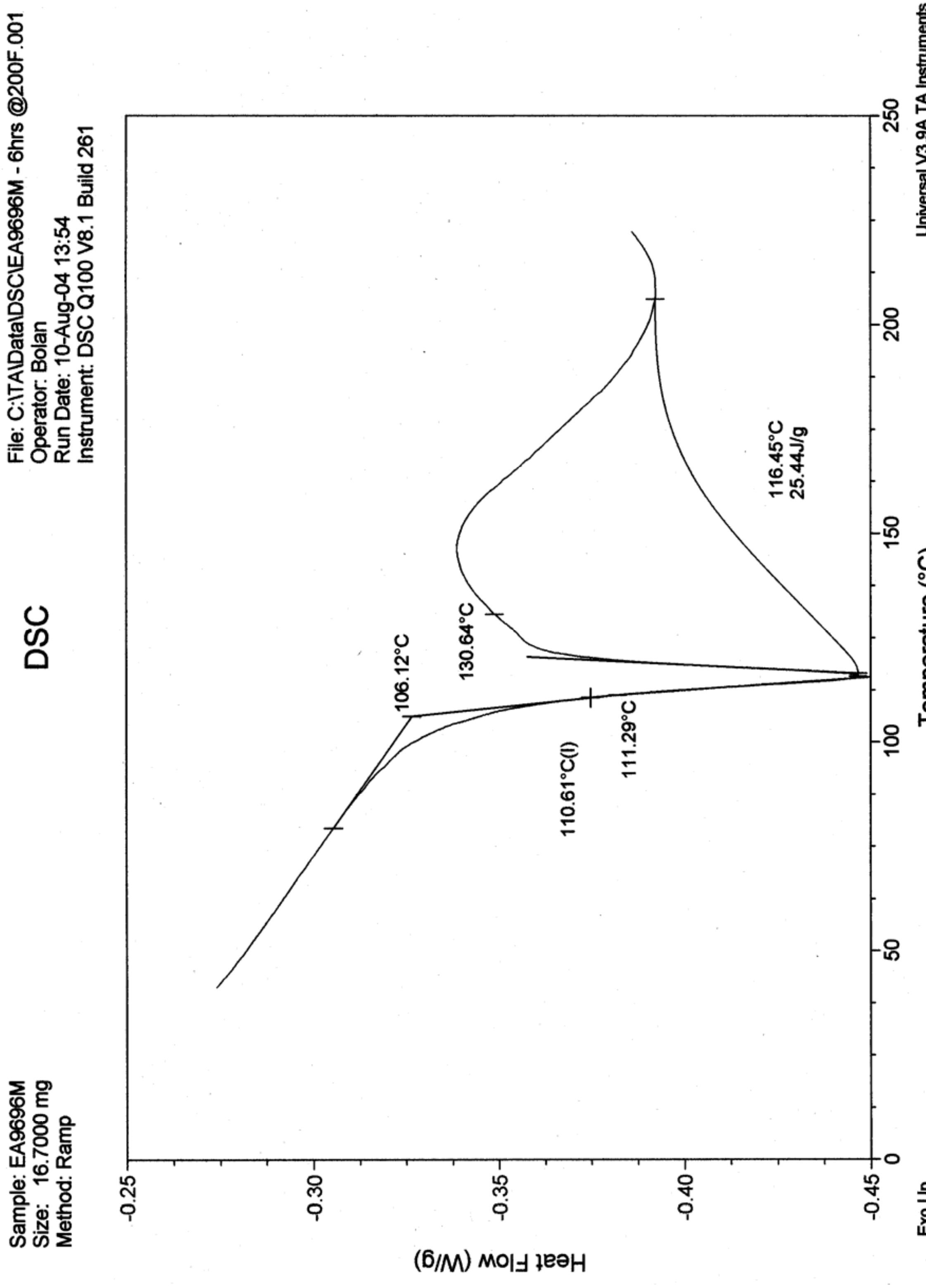


Figure A15: Energy Released During 8 Hours at 180°F for EA 9696

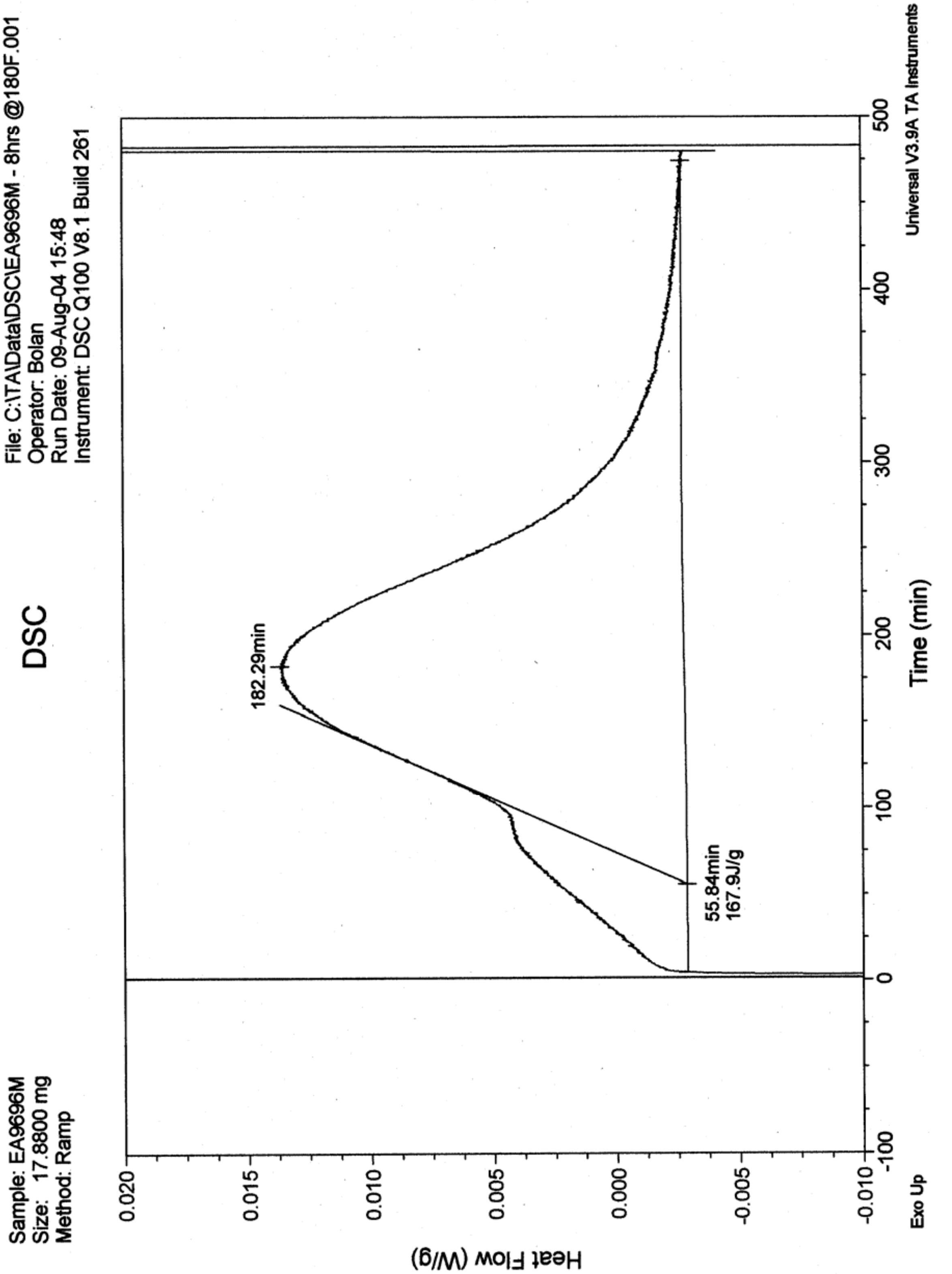


Figure A16: T_g and Residual Exotherm After 8 Hours at 180°F for EA 9696

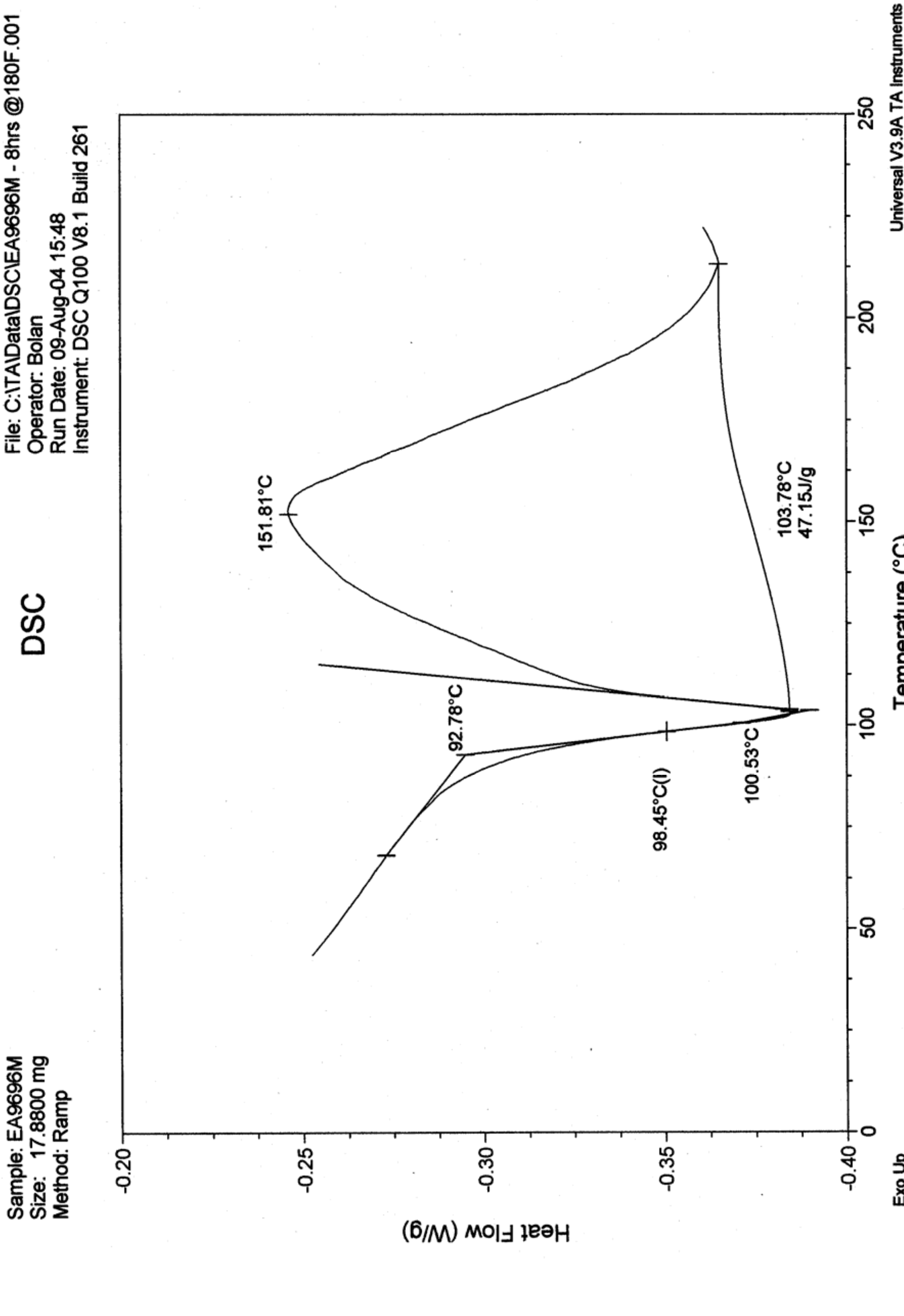


Figure A17: Energy Released During Baseline Scan (10°C/min) for EA 9628

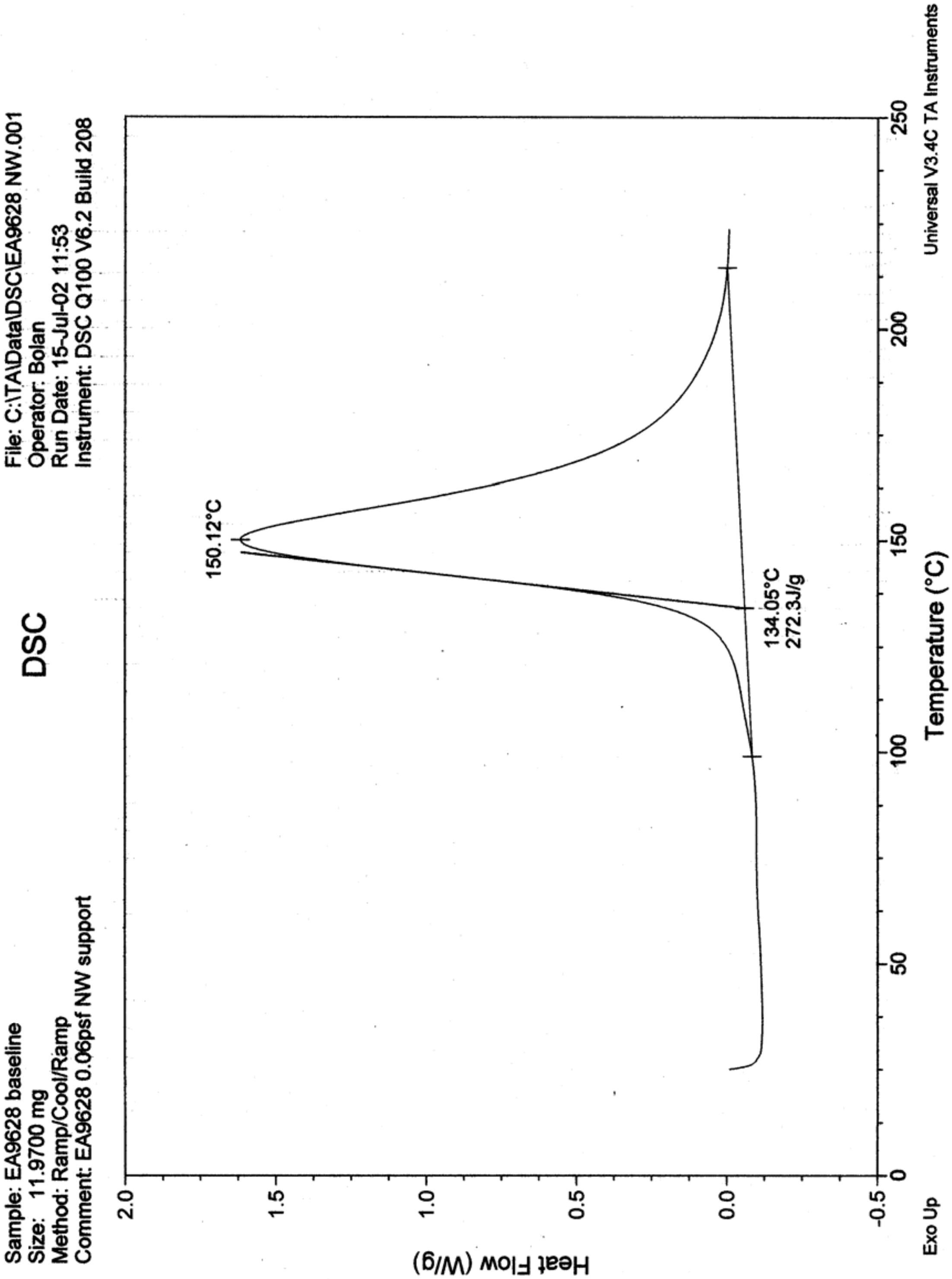


Figure A18: T_g of EA 9628 After Baseline Scan

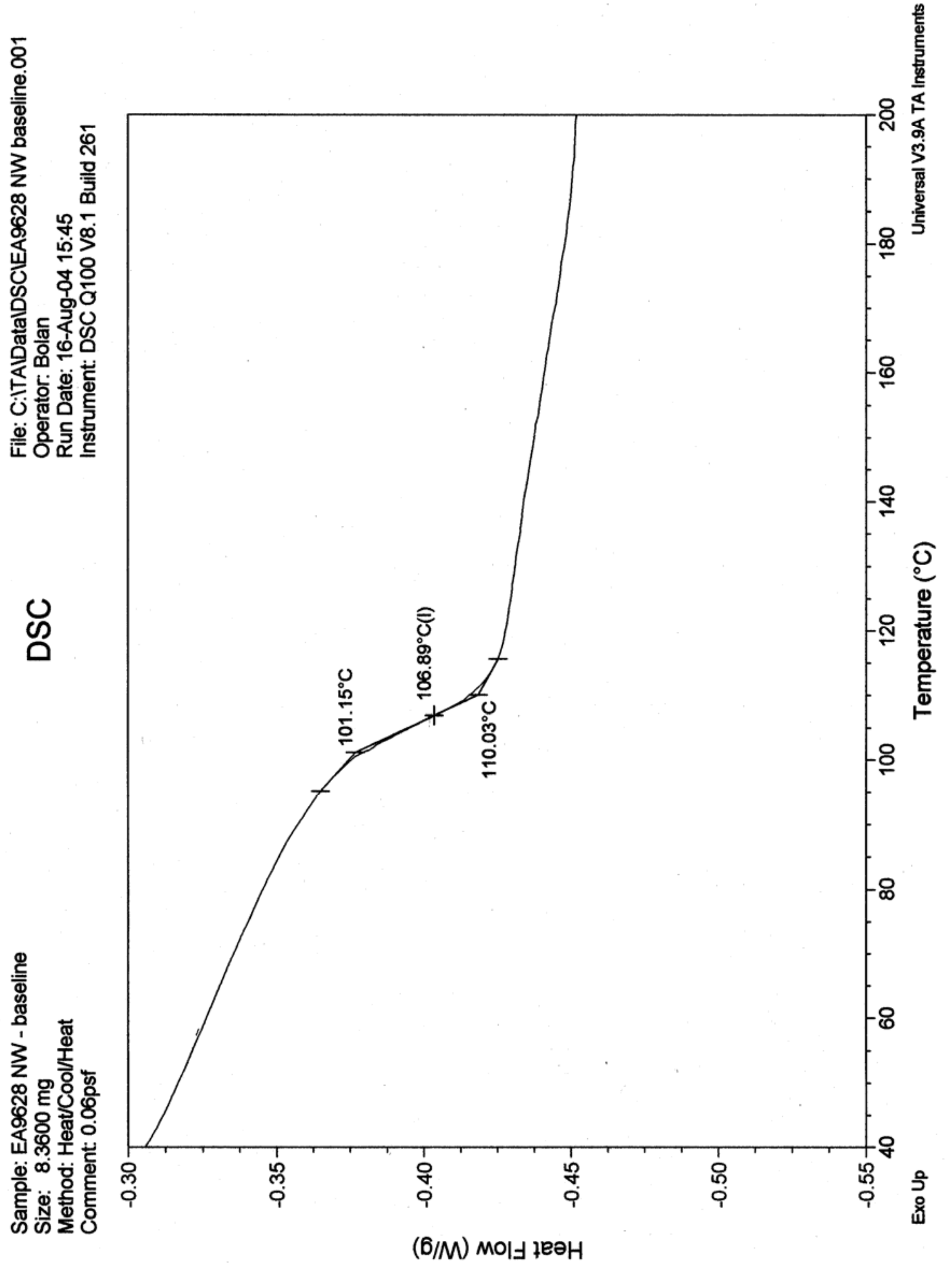


Figure A19: Energy Released During 1-Hour Cure at 250°F for EA 9628

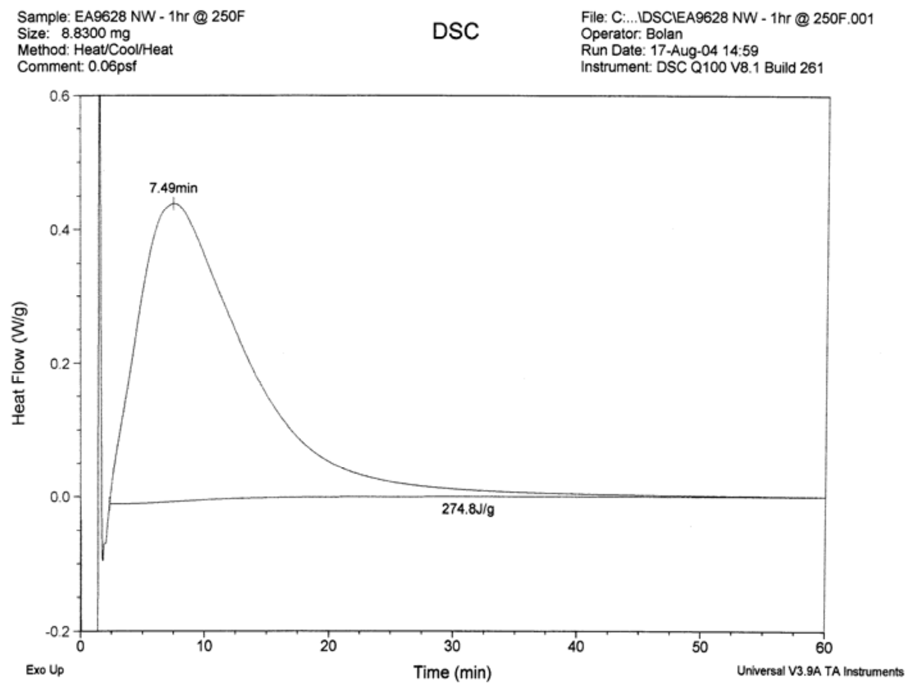


Figure A20: T_g and Residual Exotherm After 1 Hour at 250°F for EA 9628

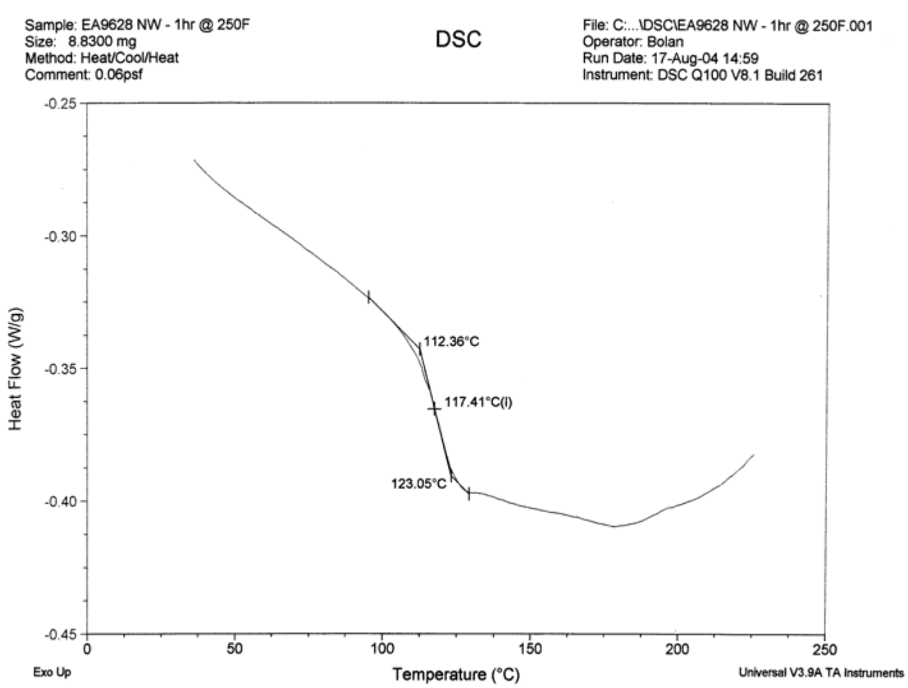


Figure A21: Energy Released During 6-Hour Cure at 200°F for EA 9628

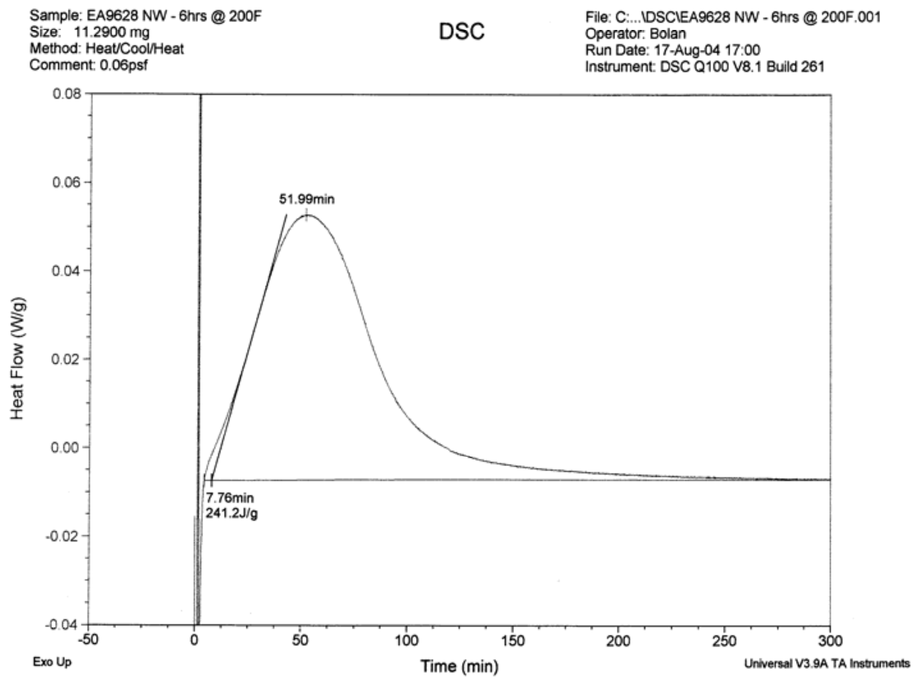


Figure A22: T_g and Residual Exotherm After 6 Hours at 200°F for EA 9628

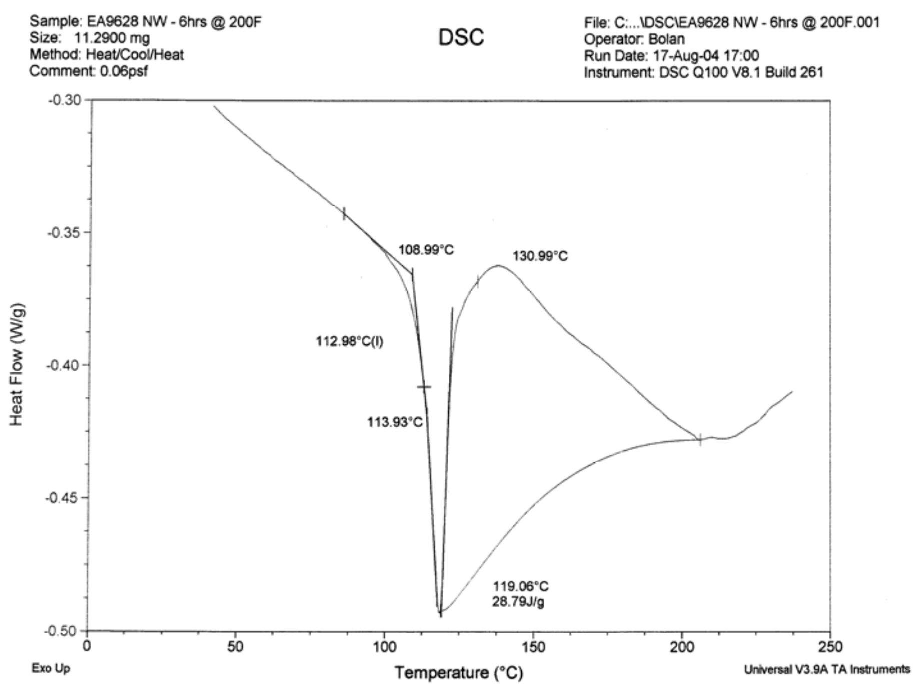


Figure A23: Energy Released During 8-Hour Cure at 180°F for EA 9628

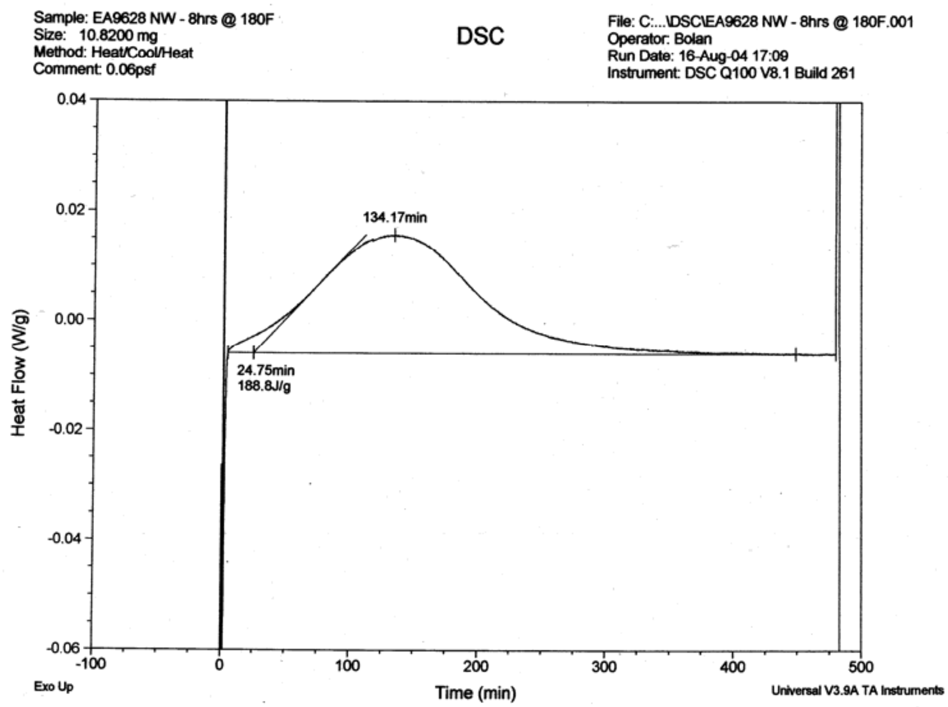


Figure A24: T_g and Residual Exotherm After 8 Hours at 180°F for EA 9628

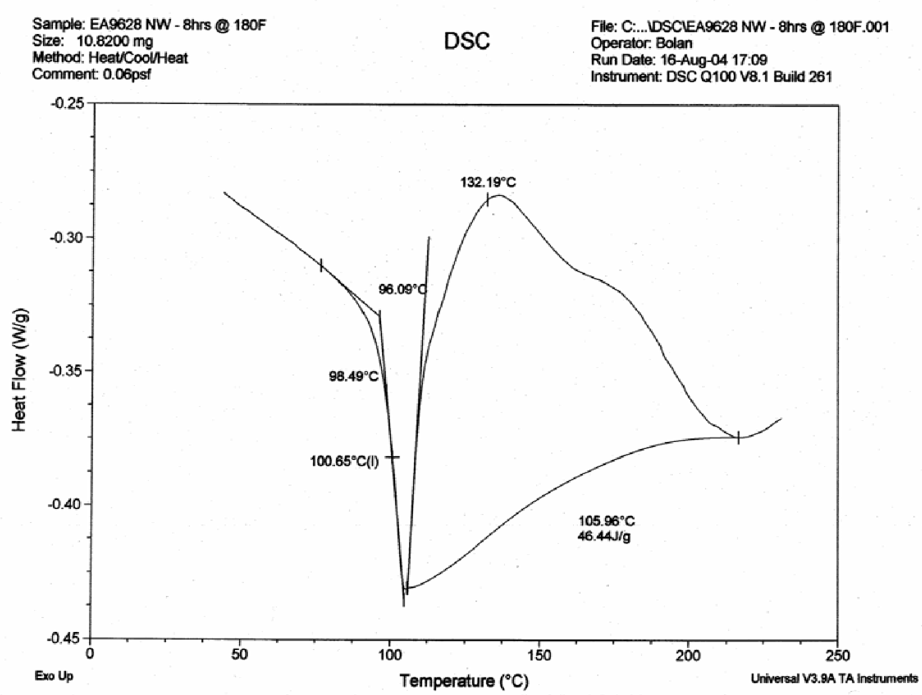


Figure A25: Energy Released During Baseline Scan (10°C/min) for FM 73M

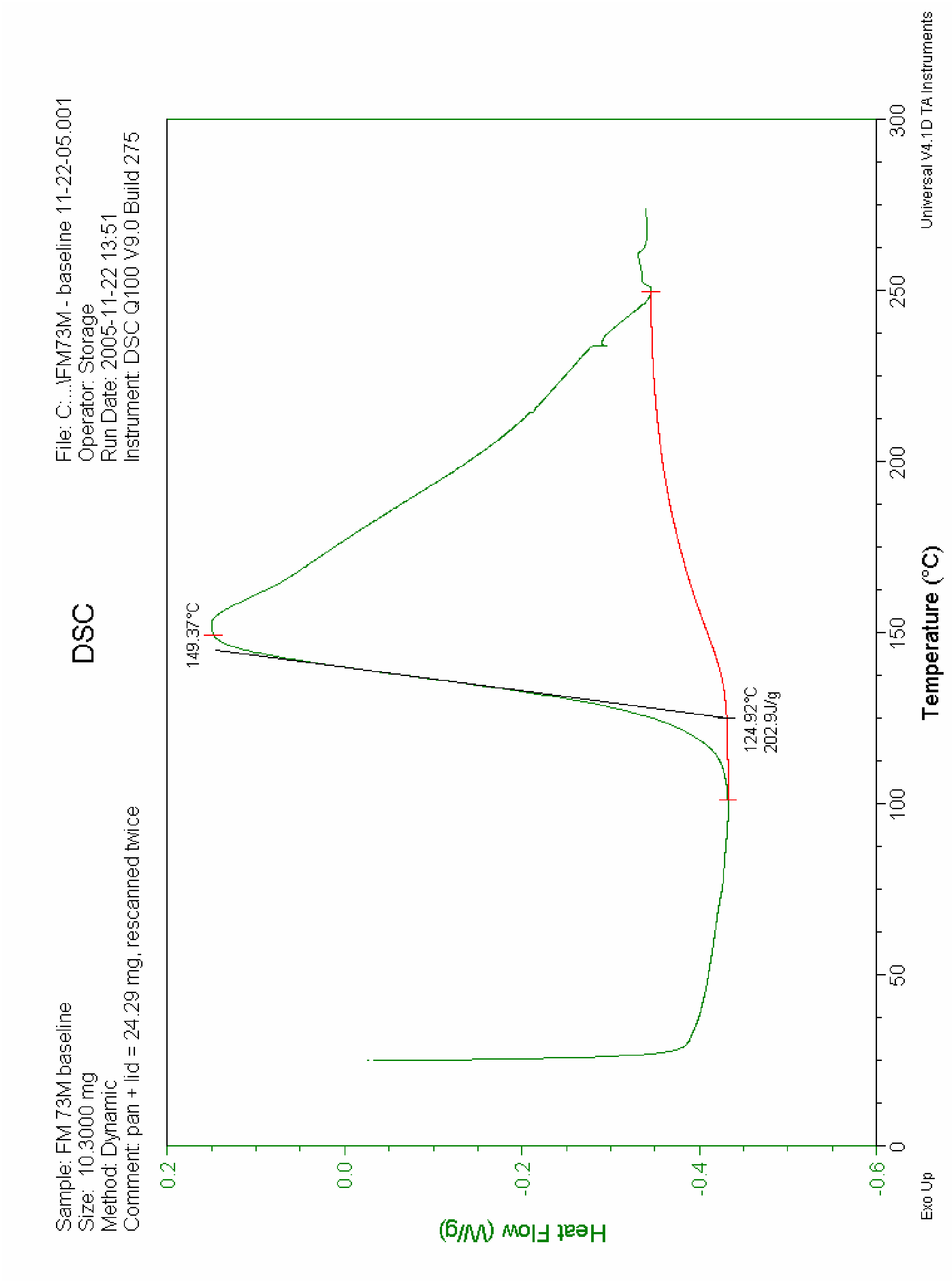


Figure A26: T_g of FM 73M after Baseline Scan

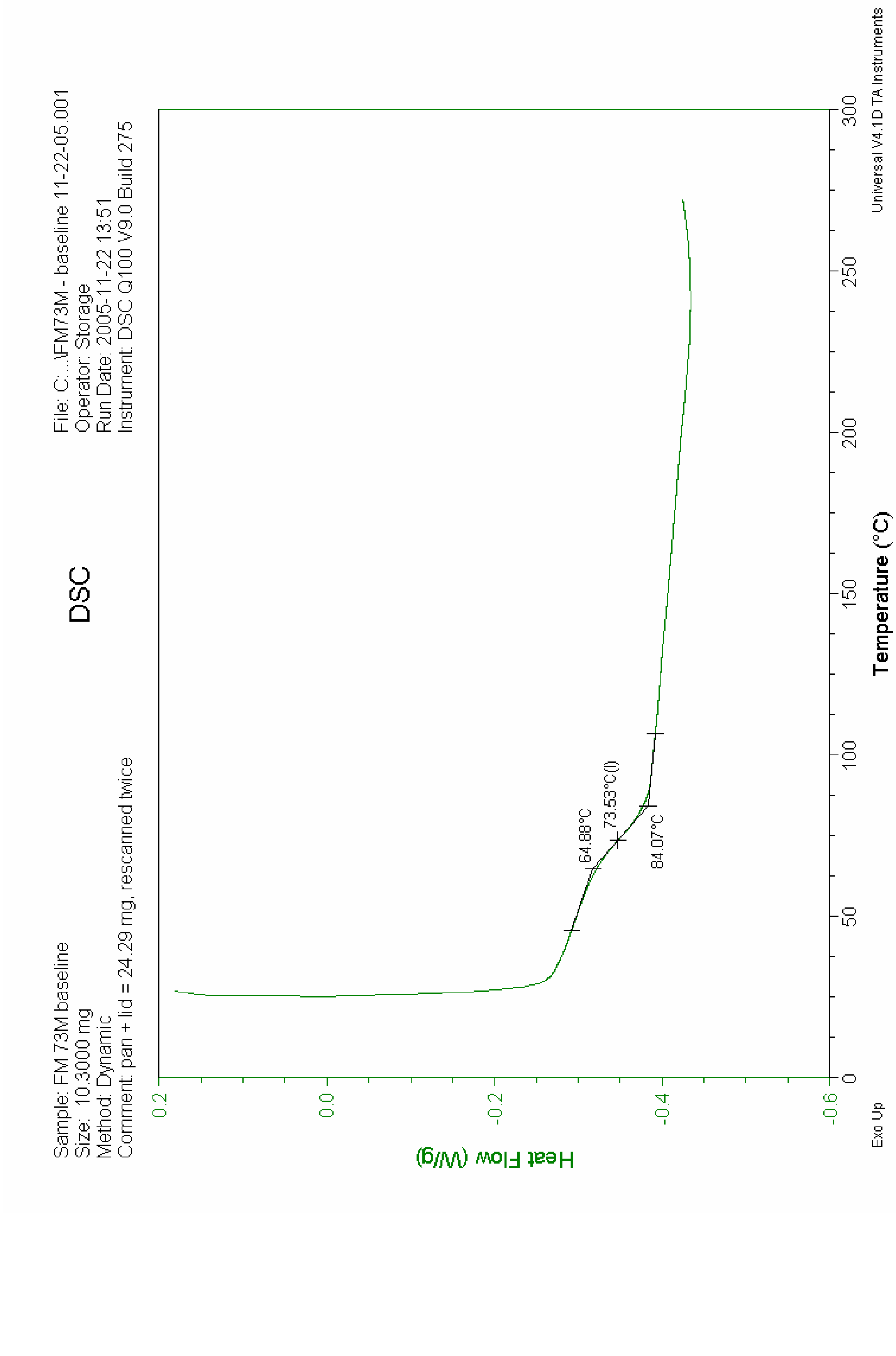


Figure A27: Energy Released During 1-Hour Cure at 250°F for FM 73M

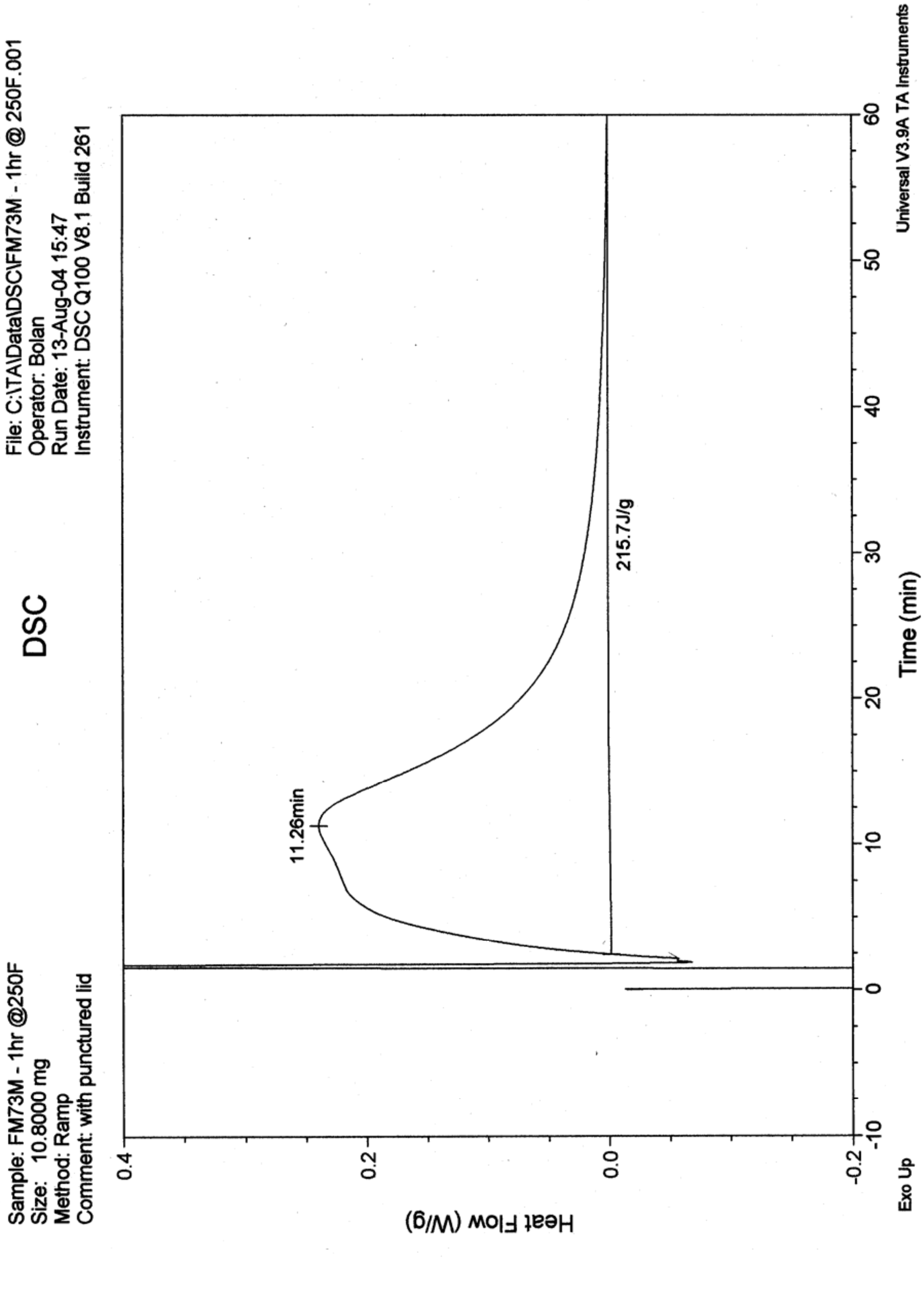


Figure A28: T_g and Residual Exotherm After 1 Hour at 250°F for FM 73M

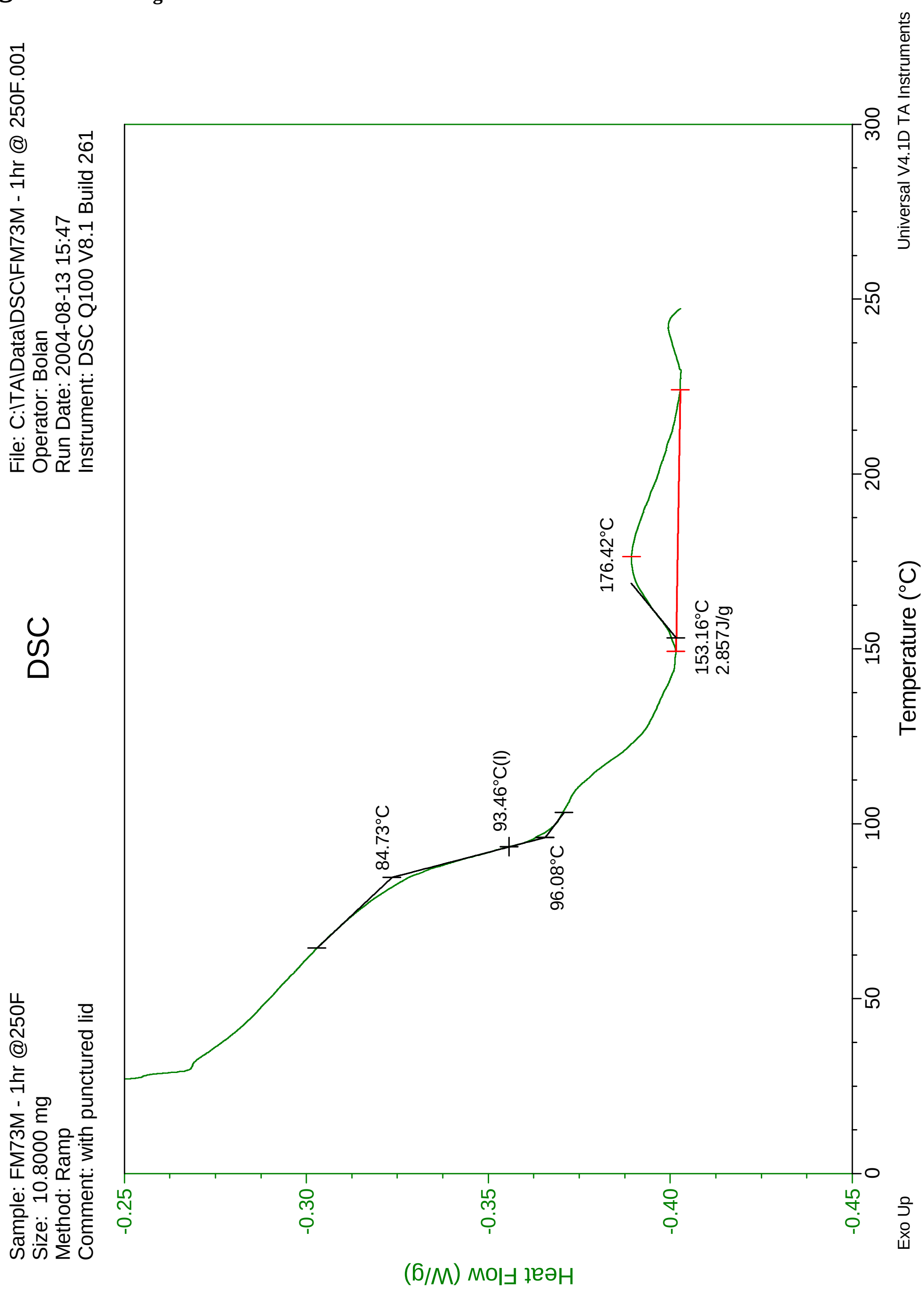


Figure A29: Energy Released During 6-Hour Cure at 200°F for FM 73M

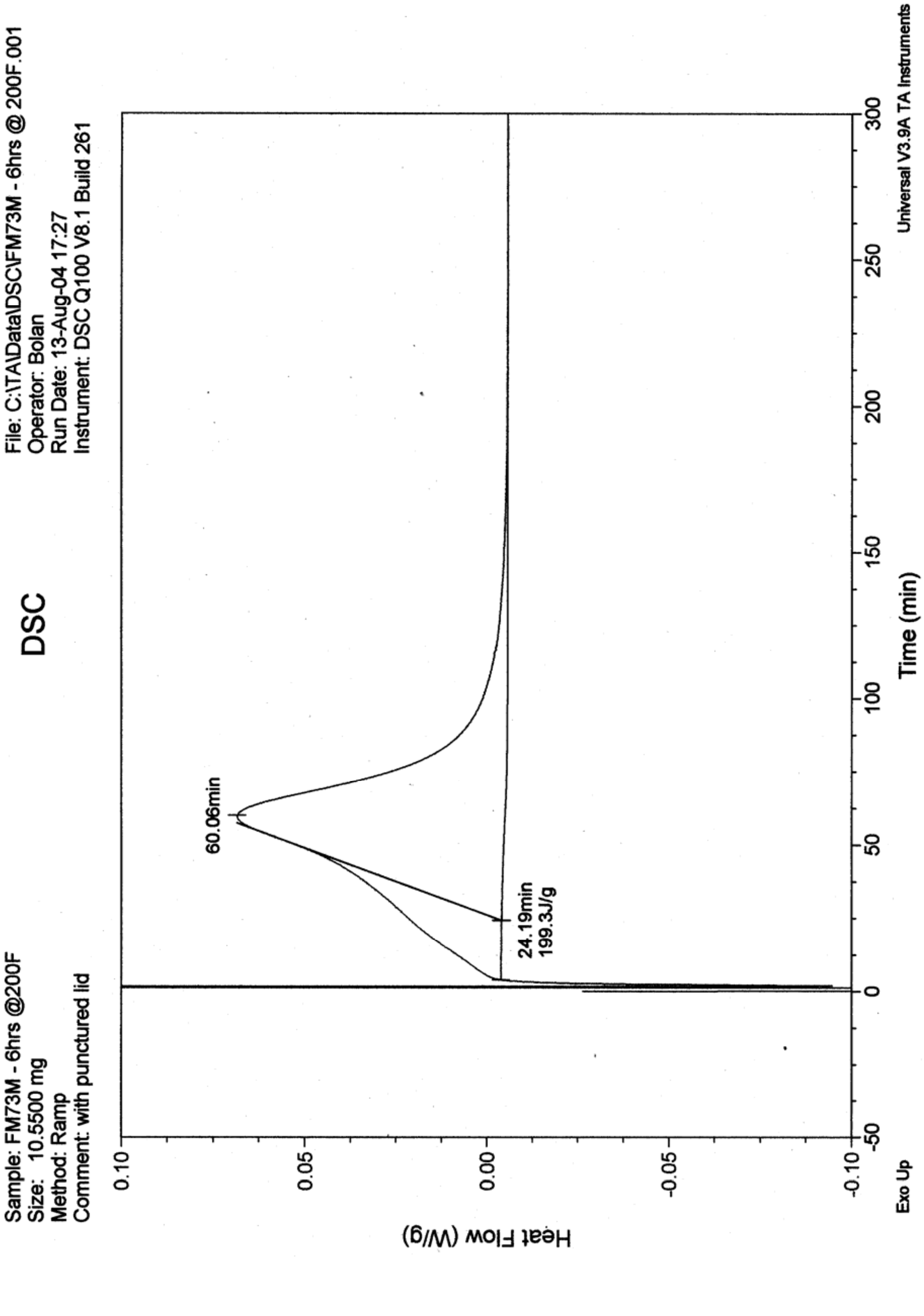


Figure A30: T_g and Residual Exotherm After 6 Hours at 200°F for FM 73M

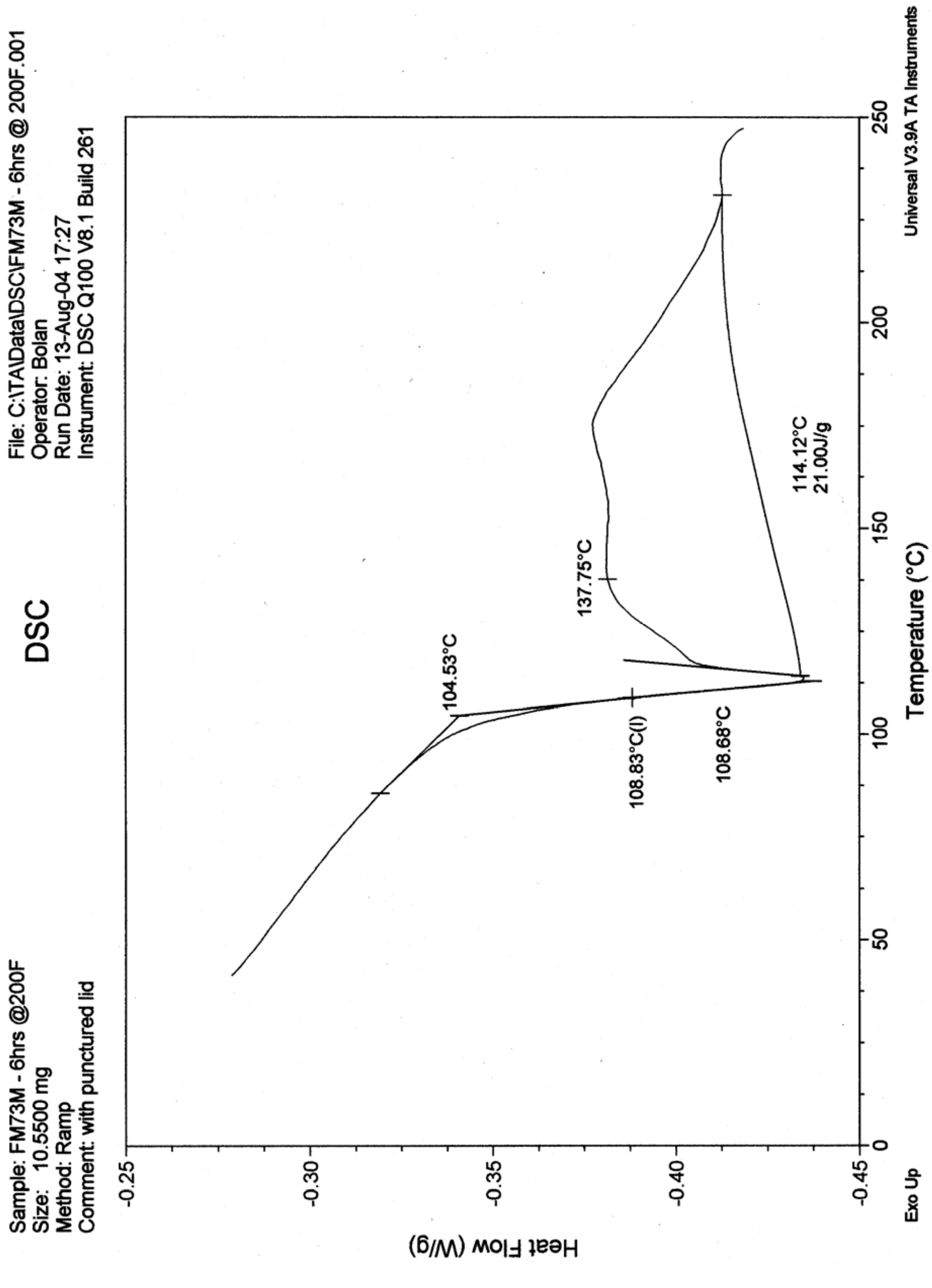


Figure A31: Energy Released During 8-Hour Cure at 180°F for FM 73M

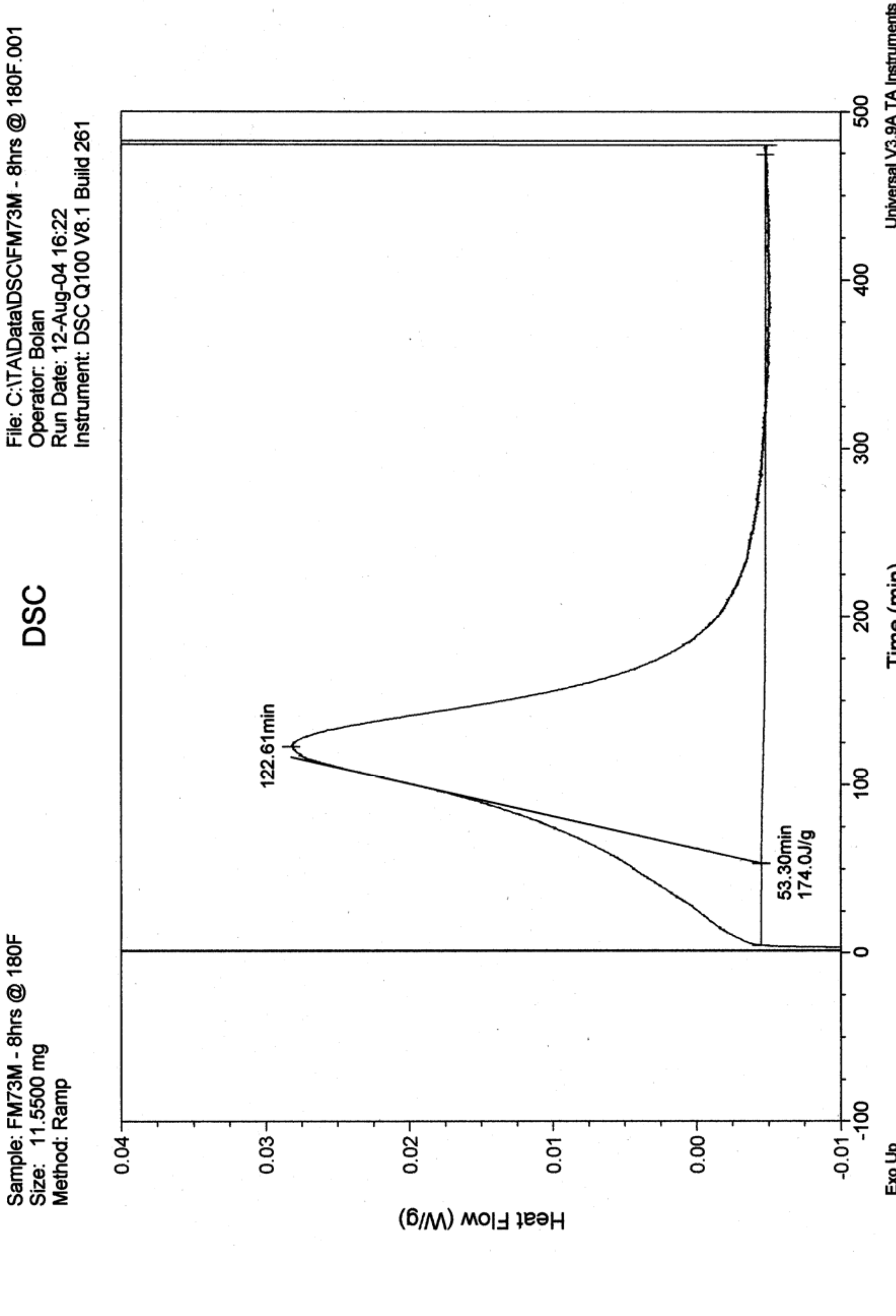
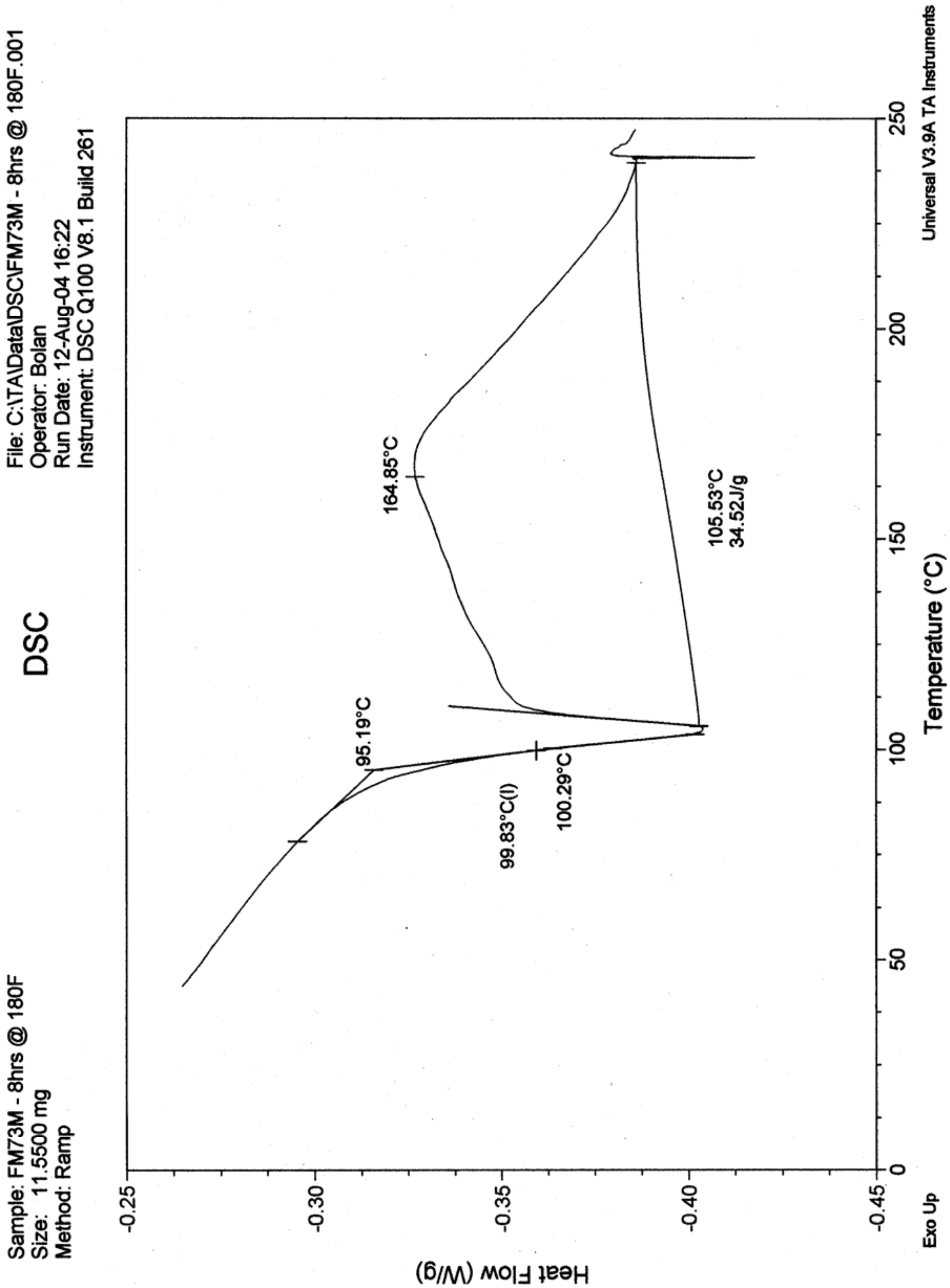


Figure A32: T_g and Residual Exotherm After 8 Hours at 180°F for FM 73M



APPENDIX B:

Mechanical Testing Results for Individual Specimens

Table B1: EA 9628 Mechanical Test Data from Table 3

Test	Cure Cycle	Test Condition	Specimen Number 1	2	3	4	5	6	Average	Std Dev
Lap Shear (psi)	1 hr @ 250°F	70°F	5935	5953	5727	5882	6080	n/a	5915	128
		180°F-Dry	4096	3976	4124	4127	4185	n/a	4102	77
		180°F-Wet	3443	3667	3376	3556	3540	n/a	3516	112
	4 hr @ 200°F	70°F	5562	5721	5529	5727	5504	n/a	5609	107
		180°F-Dry	4664	4714	4480	4408	4537	n/a	4561	127
		180°F-Wet	3430	3420	3470	3712	3932	n/a	3593	224
	8 hr @ 180°F	70°F	5057	5022	5047	5145	5015	n/a	5057	52
		180°F-Dry	3673	3588	3340	3773	3704	n/a	3616	168
		180°F-Wet	3520	3420	3482	3490	3594	n/a	3501	63
Floating Roller Peel (pli)	1 hr @ 250°F	-65°F	54.1	52.4	51.2	58.3	51	n/a	53.4	3.0
		70°F	54.7	58.5	63.4	60.8	51.9	n/a	57.9	4.6
	4 hr @ 200°F	-65°F	30.1	32.7	37.3	39.5	38.3	n/a	35.6	4.0
		70°F	32.7	36.9	38.2	38.3	36.3	n/a	36.5	2.3
	8 hr @ 180°F	-65°F	33.2	35.5	34.5	34.7	31.6	n/a	33.9	1.5
		70°F	30.0	29.6	31.0	32.1	30.5	n/a	30.6	1.0
Climbing Drum Peel (in*lb/in)	1 hr @ 250°F	70°F	17.9	17.7	18.1	18.7	16.7	16.2	17.6	0.9
	4 hr @ 200°F	70°F	9.6	9.6	10.9	11.8	12.2	13.3	11.2	1.5
	8 hr @ 180°F	70°F	8.0	8.5	9.0	9.2	8.8	9.0	8.8	0.4

n/a: not applicable, only 5 specimens per condition required
n/t: not tested, specimen damaged during setup

Table B2: EA 9696 Mechanical Test Data from Table 3

Test	Cure Cycle	Test Condition	Specimen Number 1	2	3	4	5	6	Average	Std Dev
Lap Shear (psi)	1 hr @ 250°F	70°F	5533	5616	5624	5690	5637	n/a	5620	57
		180°F-Dry	4600	4955	4753	4880	4800	n/a	4798	135
		180°F-Wet	3092	3059	3363	3135	3340	n/a	3198	143
	6 hr @ 200°F	70°F	6096	5944	5758	5830	5900	n/a	5906	128
		180°F-Dry	4402	4177	4146	4239	4300	n/a	4253	102
		180°F-Wet	3877	3750	3745	3761	3580	n/a	3743	106
	8 hr @ 180°F	70°F	5465	5320	5463	5514	5563	n/a	5465	91
		180°F-Dry	3567	3518	3488	3514	3582	n/a	3534	39
		180°F-Wet	3731	3443	3548	3640	3708	n/a	3614	119
Floating Roller Peel (pli)	1 hr @ 250°F	-65°F	66.1	64.3	55.2	48.8	48.1	n/a	56.5	8.4
		70°F	74.1	81.1	68.5	83.6	52.4	n/a	71.9	12.4
	6 hr @ 200°F	-65°F	n/t	52.8	51.4	57.1	48.0	n/a	52.3	3.8
		70°F	66.5	63.7	54.3	n/t	44.4	n/a	57.2	10.0
	8 hr @ 180°F	-65°F	42.6	49.8	49.2	49.2	40.5	n/a	46.3	4.4
		70°F	48.9	53.1	51.9	45.7	42.3	n/a	48.4	4.4
Climbing Drum Peel (in*lb/in)	1 hr @ 250°F	70°F	20.6	23.8	17.3	19.9	21.4	21.2	20.7	2.1
	6 hr @ 200°F	70°F	11.3	12.2	12.1	14.4	16.5	14.3	13.5	1.9
	8 hr @ 180°F	70°F	12.2	13.5	13.9	12.6	13.0	12.3	12.9	0.7

n/a: not applicable, only 5 specimens per condition required
n/t: not tested, specimen damaged during setup

Table B3: FM 73M Mechanical Test Data from Table 3

Test	Cure Cycle	Test Condition	Specimen Number 1	2	3	4	5	6	Average	Std Dev
Lap Shear (psi)	1 hr @ 250°F	70°F	5769	5908	5608	5835	5822	n/a	5788	112
		180°F-Dry	4280	4345	4337	4369	4312	n/a	4329	34
		180°F-Wet	3134	3184	3018	2498	2620	n/a	2891	312
	4 hr @ 200°F	70°F	6051	6075	6167	6157	6212	n/a	6132	67
		180°F-Dry	4416	4134	4062	4250	3946	n/a	4162	180
		180°F-Wet	3602	3404	3367	3400	3447	n/a	3444	93
	7 hr @ 180°F	70°F	6384	6018	5976	6294	6185	n/a	6171	175
		180°F-Dry	4169	4521	4144	4239	3943	n/a	4203	209
		180°F-Wet	3931	3410	3348	3444	3382	n/a	3503	242
Floating Roller Peel (pli)	1 hr @ 250°F	-65°F	67.8	73.4	72.3	71.8	55.4	n/a	68.1	7.4
		70°F	84.9	93.5	91.7	90.4	83.8	n/a	88.9	4.3
	4 hr @ 200°F	-65°F	n/t	37.9	34.8	34.6	36.8	n/a	36.0	1.6
		70°F	42.9	49.0	50.8	48.4	47.4	n/a	47.7	3.0
	7 hr @ 180°F	-65°F	35.2	30.0	26.6	29.9	33.7	n/a	31.1	3.4
		70°F	41.4	42.7	46.5	40.9	41.5	n/a	42.6	2.3
Climbing Drum Peel (in*lb/in)	1 hr @ 250°F	70°F	19.7	22.2	n/t	24.5	23.8	24.2	22.9	2.0
	4 hr @ 200°F	70°F	14.4	13.3	15.2	14.5	12.9	14.0	14.1	0.8
	7 hr @ 180°F	70°F	11.7	11.3	12.5	12.0	11.8	11.2	11.8	0.5

n/a: not applicable, only 5 specimens per condition required
n/t: not tested, specimen damaged during setup

Table B4: AF 163-2M Mechanical Test Data from Table 3

Test	Cure Cycle	Test Condition	Specimen Number 1	2	3	4	5	6	Average	Std Dev
Lap Shear (psi)	1 hr @ 250°F	70°F	5737	5600	5613	5900	5671	n/a	5704	122
		180°F-Dry	3892	3827	3800	3847	3658	n/a	3805	89
		180°F-Wet	2706	2631	2969	2935	2906	n/a	2829	151
	6 hr @ 200°F	70°F	5363	5667	5712	5724	5861	n/a	5665	184
		180°F-Dry	n/t	3867	3730	3788	3757	n/a	3786	59
		180°F-Wet	3072	2978	2908	3058	3104	n/a	3024	80
	8 hr @ 180°F	70°F	5200	5322	5300	5227	5129	n/a	5236	78
		180°F-Dry	2828	2755	2794	3006	2806	n/a	2838	98
		180°F-Wet	3186	2953	2873	2888	3027	n/a	2985	128
Floating Roller Peel (pli)	1 hr @ 250°F	-65°F	68.9	63.9	64.3	73.6	73.5	n/a	68.8	4.7
		70°F	77.0	80.2	79.1	78.7	73.0	n/a	77.6	2.8
	6 hr @ 200°F	-65°F	63.6	60.0	56.7	60.3	56.3	n/a	59.4	3.0
		70°F	65.3	66.4	64.4	62.0	62.5	n/a	64.1	1.9
	8 hr @ 180°F	-65°F	55.3	63.3	55.3	51.9	45.9	n/a	54.3	6.3
		70°F	61.8	61.9	63.1	62.3	60.7	n/a	62.0	0.9
Climbing Drum Peel (in*lb/in)	1 hr @ 250°F	70°F	17.3	17.7	18.7	20.2	17.0	17.2	18.0	1.2
	6 hr @ 200°F	70°F	14.0	13.6	14.7	16.0	16.3	16.0	15.1	1.2
	8 hr @ 180°F	70°F	14.0	16.6	14.3	16.2	16.5	16.0	15.6	1.1

n/a: not applicable, only 5 specimens per condition required
n/t: not tested, specimen damaged during setup